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THERMAL-GENERATED AEROSOLS FOR CONTROL OF BITING FLIES IN CANADA[1]

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INTRODUCTION

Field experiments were conducted to determine the effectiveness of thermal-generated aerosols in the control of adult mosquitoes and black flies in northern Canada. These tests, with a heavy ground aerosol generator[3], were carried out at Whitehorse, Y.T., during the summer season of 1949, and at Camp Borden, Ontario, and Fort Churchill, Manitoba, in 1950.

The early experimental work on aerosols for the control of agricultural pests has been reviewed by Latta (9). Brescia (1) described the effectiveness of a DDT aerosol in the control of salt-marsh species of mosquito and of anopheline mosquitoes, in open and forested areas. The promising performance of the generator used in this investigation was reported first in 1945 by Vorhies and Wehrle (17), and in 1946 by Glasgow and Collins (4). The value of ground-applied aerosols and of this generator in particular in controlling adult biting insects, under conditions similar to those encountered during the present study, was assessed by Wilson *et al.* (18). They reported that satisfactory control was not maintained for periods longer than 24 hours, even though the biting rate was low immediately after the application. They also observed that the aerosol dispersed erratically in wooded areas, drifted along roads, and often was dissipated over the trees. However, Latta (10) reported that he overcame these difficulties and that an effective dispersal in forest areas is possible.

DROPLET SIZE AND DENSITY

The importance of the relation of droplet size to the effective distance and toxicity of an aerosol has been emphasized by Gibbs (3), Sell (15), Brescia (1), Goodhue and Riley (7), Potts (14), Latta *et al.* (11), and Yeomans *et al.* (19). It has been established that to be most effective against adult mosquitoes an aerosol should have a mass median diameter of 10 to 20 microns, and be borne by air moving at 1 to 4 miles per hour, during an inversion. However, Latta (10) reported that a steady wind direction is the main prerequisite for the successful treatment of forest areas, as the turbulence within the forest distributes the aerosol to all

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[3] Todd Insecticidal Fog Applicator (TIFA).

heights and yet maintains a general downwind movement. Hence, in forested areas, the restriction of use of ground-applied aerosols to sunset and sunrise hours, when an inversion normally prevails, has been removed.

In the light of the above information this study was designed to determine the most effective DDT formulation and the maximum distance of effective dispersal, including a physical study of the behaviour of the aerosol cloud in relation to its effect on the insects. Experimental variation was limited to amount applied per foot of frontage and to aerosol density during conditions of zero or negative temperature gradient (inversion). Aerosol density is dependent only on the rate of emission and the speed of movement of the vehicle transporting the generator, whereas amount applied is also dependent on the concentration of the insecticide used.

The procedure outlined included, first, the calibration of the generator for the output of each formulation to produce an aerosol of 10-20 microns mass median diameter; and second, observations on changes in the physical characteristics of the aerosol clouds as they were carried downwind. The latter included droplet sizes and concentrations of the aerosols.

Materials and Methods

Two models of the generator were used: Model No. 40-E-47 in 1949, and Model No. 40-E-48 in 1950. The latter was equipped with a thermocouple for measuring the temperature of the air blast to which the insecticide was subjected; this is a critical factor in the determination of the droplet spectrum. With the model used in 1949 it was necessary to assume that it was directly proportional to the pressure at which fuel was supplied to the combustion chamber that heated the air, and that this temperature remained constant during the period of operation.

Aerosol samples were obtained on glass microscope slides coated with an oleophobic silicone (G. E. Drifilm No. 9987). Cascade impactors (Casella's model of K. R. May's design) were adopted for sampling in the 1950 season. In this case the sample was impinged on a series of four coated slides, by being drawn through progressively finer jets within an airtight system. An assembly was constructed to enable these impactors to be used in the field and consisted of a 25-gallon drum on which the impactor and a 6-inch × 6-inch copper screen cylinder for caging insects was supported on a steel rod. Air was drawn from the impactor, through a calibrated needle valve, into the evacuated drum.

Droplet measurements were made with a microscope fitted with an ocular micrometer that was later replaced by the Portion modification of the Patterson-Cawood graticule described by May (13).

Each TIFA machine was first calibrated for the output that would produce aerosols with mass median diameters of 10 and 20 microns with the formulations to be used. The output at each setting of the particle size selector, at a constant formulation pressure of 20 p.s.i., was measured by disconnecting the insecticide hose at the head. Samples of the aerosol produced at each of these settings were then taken. In the first phase these samples were obtained by waving a coated slide in the aerosol stream, 2 yards from the nozzle of a stationary generator. Controls ensured that the droplet spectrum did not vary over a short period of operation because of an increase in the temperature of the air blast.

In the second phase the cascade impactor assemblies were placed 25 yards from a stationary generator or from an emission line. A sample was obtained at an air intake of 10 or 17.5 litres per minute for a period estimated by the operator from the apparent density of the aerosol cloud at that point. The same procedure was followed during field biological experiments.

The droplets were assessed as soon as possible after the sample had been obtained. The number and size of the droplets in the centre square inch of a number of open slides and of at least 200 droplets on each of the four impactor slides were tabulated. The spreading coefficient (ratio of diameter of droplet to diameter of observed lens) was computed by May's focal length method (13). The cumulative percentage volume in each droplet size was plotted as cumulative frequency on logarithmic-probability paper; from the straight line so obtained for the sample, the median diameter by volume (i.e., the mass median diameter) could be read.

The initial plan for observing the physical characteristics of aerosols in the field included the suspension of coated slides in their paths, and the placing of Petri dishes at each of these points to collect any droplets falling out of the aerosol cloud. These slides yielded no samples, and available facilities did not permit an accurate examination of the minute deposits in the Petri dishes. The suspended slides were replaced in the second season by three impactors containing uncoated slides placed at various distances from the base line. The impactors were operated at 17.5 litres per minute for 3 minutes, which was sufficient to cover the passage of the aerosol cloud. Each sample was washed from the set of slides for an accurate determination of the DDT content. This would provide data on the density of the aerosol that could be related to its observed toxicity. After the generator had been calibrated for the output of the formulations, observations were made on the physical characteristics of the aerosol clouds as they were carried downwind. Samples were taken in cascade impactors set up at distances of 200, 300, 600, and 700 yards downwind of the machine.

Results

Table 1 shows the relation of air-blast temperature as determined by the fuel pressure and of delivery rate as determined by the particle size selector to the droplet spectrum and its mass median diameter. The figures indicate that the increase in droplet size with a higher delivery rate may be cancelled by a corresponding increase in air-blast temperature; thus the aerosol output can be increased while the droplet spectrum is maintained within the desired range. However, this approach was not carried further, as the extent of thermal destruction of DDT and the safety limits of the generator were not known.

The data indicate a significant difference between the outputs of the formulations based on the lighter oils and of those based on the heavier oils, S.A.E. 10 and Diol 55. The greater delivery rate with the lighter oils is due mainly to their lower viscosity, but other factors such as density and surface tension may be involved. The second point of interest is the relatively narrow limits of the range of droplet sizes given by fuel oil and S.A.E. 10 oil.

TABLE 1.—THE RELATION OF OUTPUT AND DROPLET SIZE FOR TIFA MODEL NO. 40-E-47 (FORMULATION PRESSURE OF 20 P.S.I.)

Formulation*	Fuel pressure (p.s.i.)	Output		Droplet size**	
		Selector setting	Rate gal./hr.	Mass median diameter microns	Range microns
5 per cent DDT (wt./wt.) in fuel oil No. 2	50	16	—	4.1	1–10
		18	—	7.3	1–14
		20	20.0	14.9	1–26
		21	23.7	26.3	1–39
		22	25.0	25.2	4–41
		23	28.1	—	—
		24	31.0	31.3	9–50
	60	20	20.0	13.1	1–19
		22	25.0	13.7	1–26
		24	31.0	13.8	9–27
	70	24	31.0	8.8	1–16
		25	—	16.6	3–50
20 per cent DDT (wt./wt.) in S.A.E. 10 oil	50	14	—	11.1	1–18
		15	8.3	14.5	1–26
		16	—	14.4	1–27
		18	12.9	23.4	1–37
		20	—	29.4	9–37
	60	16	—	9.4	1–18
		18	12.9	15.2	1–26
		20	—	19.6	9–27
	70	22	—	30.4	3–46
20 per cent DDT (wt./wt.) in Mentor 29	50	18	16.9	8.9	4–18
		20	17.3	22.3	4–55
	60	20	17.3	16.7	4–40
	70	20	17.3	2.3	1–5
		22	22.5	21.8	3–39
20 per cent DDT (wt./wt.) in Diol 55	50	16	—	4.6	1–9
		18	11.8	17.2	5–55
		20	15.0	38.7	5–64
	60	18	11.8	9.8	1–23
	70	20	15.0	19.8	4–32

* Prepared from a stock solution of 35 per cent DDT (wt./wt.) in Velsicol AR 50.
** The arithmetic mean of the mass median diameters determined for a series of samples.

The rate of output from the second generator was obtained in 1950 for a 5 per cent (wt./wt.) solution of technical DDT in kerosene and fuel oil No. 2. The range for the kerosene was from 0.77 to 45.0 gallons per hour. The spread factor for this solution on G. E. Drifilm was 0.525, based on a refractive index of 1.4598 at 20° C.

The samples obtained in the cascade impactors that were placed at distances of 200 to 700 yards downwind of the machine did not represent the true droplet spectrum, because of the failure of the impactors to take

in the larger droplets, the presence of which at these distances could be readily demonstrated by waving coated slides in the aerosol clouds. Raising the impactor intake from 10 litres/min. to 17.5 litres/min. did not remedy the situation. In a total of 12 samples so taken, the mass median diameters varied between 3.4 and 9.4 microns, and showed no correlation with delivery rate of the machine or sampling rate of the impactor.

Discussion

The calibration of the generator is the first requirement in the field use of thermal-generated aerosols, because the production of a known droplet spectrum is essential. This generator and others examined are not calibrated by the manufacturers to the degree that mosquito control projects, and particularly assessments, require. The attainment of an accurate assessment of the droplet spectrum occurring in the aerosol cloud was the problem faced and not satisfactorily solved in this investigation. However, the results did indicate the significance of the solvent in the density of the aerosol produced. That is, a solution based on fuel oil No. 2 has a much higher output for a droplet spectrum of 15 microns than one based on S.A.E. 10 oil, and, therefore, the density of the aerosol of the former is much greater. The optimum or minimum density for effective control is not known; but when it is known, the choice of the fog oil depends not only on its ability to dissolve large quantities of DDT but also on the droplet spectrum produced from it.

BIOLOGICAL ASSESSMENT

This phase of the investigation was designed to assess the value of ground-applied thermal aerosols in protecting localized areas against biting flies. These areas may be surrounded by dense or open forest, or by prairie or tundra, and each presents its own problems. It has been observed previously that biting flies in an inhabited area can be controlled by frequent applications of an aerosol applied by standard techniques. It was proposed to use these techniques and any innovations that might be developed.

Assessments of the effect of applications under forest conditions were made at Whitehorse, Y.T., in 1949, and of those in open areas at Camp Borden, Ontario, in 1950. It was desired to make biological assessments of applications carried out by the most satisfactory techniques under optimum meteorological conditions.

Materials and Methods

At Whitehorse 4 test plots were selected. Many otherwise ideal plots were inaccessible or unsuitable because of the small number of roadways and the almost constant south wind in the valley.

Plot A was on rising ground, lightly wooded with pine and aspen for 500 yards from the base line, and more densely wooded beyond, including willow and heavier undergrowth; a secondary road provided an east-west base line of 1,200 yards.

Plot B was on flat ground, more uniformly covered with pine, aspen, and willow, and rose sharply on its northern border; a small lake and grassy marsh lay in the centre of the plot. Two base lines were available, the first common to plots A and B, and the second on the western, 700-yard border.

Plot C was an isolated, rectangular area, 700 by 500 yards, on the northern border of the townsite; this bore a light growth of pine and aspen.

Plot D was undulating ground, rising sharply at the base line and bearing young pines; it was bordered on two sides by roadways, one of which formed an east-west base line of 700 yards.

At Camp Borden, with its wide expanse of open, flat pasture land, interspersed with dense stands of poplar, willow, and conifers, there were excellent and almost unlimited assessment areas.

Plot E, on Salisbury Plains, was a flat, grass-covered sandy plain.

Plot F was on flat ground densely covered with willow, poplar, and pine to a depth of 1,500 yards. "Bog Road" formed a base line running east-west for 500 yards.

Plot G was flat ground with conditions similar to those in Plot F. This had a north-south base line of 700 yards along a narrow trail.

In each test plot a check line placed more or less at right angles to the base line, flexible enough to allow for variation in wind direction, was marked with flags at 100-yard intervals. Before each test, personnel were placed at intervals along the check line to record the landing and biting rates of mosquitoes and, wherever possible, of black flies. In addition, control counts were usually taken in an adjacent, untreated area. Counts were also made 24 and 48 hours after the applications. Infiltration from the perimeters of these relatively small test plots was usually rapid.

Wire screen cages, as described in a previous section, were used at Camp Borden as a part of the cascade impactor assemblies. Adult mosquitoes captured in or near the test plot were placed in control and test cages a few minutes before the assessment. The flies were transferred to larger, cheesecloth cages immediately after the treatment. However, the mortality rate of the mosquitoes exposed in these cages to the aerosol was negligible, and no data were obtained from this method.

The predominant mosquitoes at Whitehorse, Y.T., during the assessments included *Aedes punctor* (Kby.), *A. pionips* Dyar, *A. communis* (DeG.), and *A. cataphylla* Dyar. Various species of black flies (Simuliidae) occur in this region, but those involved in the aerosol studies were not identified. The mosquitoes against which the assessments at Camp Borden were conducted included *A. intrudens* Dyar, *A. trichurus* (Dyar), and *A. stimulans* (Wlk.).

A 5 per cent (wt./wt.) solution of technical DDT was applied in all the field assessments except one. In this case, the solution was 20 per cent (wt./wt.) in S.A.E. 10 oil. The carrier was domestic fuel oil at Whitehorse, Y.T., and kerosene at Camp Borden. These solutions were applied from a TIFA generator moving along a base line at a rate of 1 to 2 m.p.h. The output was determined by the selection of the median particle diameter. However, as even an approximate particle size assessment was not completed for the generator used in 1950, the output was estimated from information contained in the TIFA operating manual.

Wind velocity was measured with a Biram's anemometer, and small smoke generators indicated wind direction and stability.

Many of the field experiments were terminated or results from them were invalidated by unexpected changes in wind direction and other meteorological conditions. These are not included in Table 2.

TABLE 2.—FIELD ASSESSMENT OF GROUND-APPLIED AEROSOLS AT WHITEHORSE, Y.T., 1949, AND CAMP BORDEN, ONT., 1950

Test No.	Plot	Time of test	Exterior wind direction and velocity (m.p.h.)	Output (gal./hr.)	DDT dosage per foot frontage (C) (lb.)	Mass median diameter of droplets (microns)	Droplet density per foot frontage (D)*	Distance of complete reduction (yd.)	Distance of noticeable effect (yd.)
1	B	1930	SSW1-4	20.0	8.05×10^{-4}	15	6.48×10^{6}	800	—
2	A	1940	SE2	20.0	8.05×10^{-4}	15	6.48×10^{6}	100	100
3	D	2030	S1	51.0	20.50×10^{-4}	50	0.26×10^{6}	200	400
4	A	1850	SE8	17.5**	28.10×10^{-4}	30	0.73×10^{6}	100	100
5	C	2030	S8	35.0	14.08×10^{-4}	35	0.89×10^{6}	300	500
6	A	1930	SSE6	28.0	13.05×10^{-4}	27	1.59×10^{6}	400	—
7	G	1600	W2	20.0	8.05×10^{-4}	20†	2.74×10^{6}	50	100
8	F	1600	S5	20.0	8.05×10^{-4}	20†	2.74×10^{6}	50	200
9	E	2115	W3-5	20.0	8.05×10^{-4}	20†	2.74×10^{6}	50	200
10	E	2050	W6	20.0	8.05×10^{-4}	20†	2.74×10^{6}	50	100
11	E	2050	NW3	20.0	8.05×10^{-4}	20†	2.74×10^{6}	100	100

* An approximation calculated from the mass median diameter and output, for comparative purposes only.
** Twenty per cent (wt./wt.) solution of technical DDT in S.A.E. 10 oil.
† Estimation from TIFA operating manual.

At Fort Churchill, Manitoba, the use of the generators for control measures restricted opportunities for field experiments. However, it was observed during the control operations that the aerosol could be applied at almost any hour of the day, because the cool, damp conditions were favourable to an excellent dispersal of the aerosol, which remained close to the ground.

Results and Discussion

These experiments demonstrated the practicability of thermal-generated aerosols in controlling biting-fly infestations in forested areas. The selection of suitable meteorological conditions overcame the erratic dispersal of the aerosol reported by Wilson *et al.* (18). The second objection raised by these authors was that the degree of control in the forest deteriorated within 24 hours of the application. It is felt that rapid reinfestation of such a limited area is to be expected, but that it does not detract from the value of the technique in the control plan.

An examination of the technique is now possible. Toxicity is related to droplet size and, within defined limits, is directly proportional to concentration and time of exposure of the insects to the insecticide. The expression *Ct* is often used. There are two aspects of the term *concentration* that must be considered in this investigation, namely, the concentration of the insecticide, and the concentration of the aerosol droplets in the air. To avoid confusion the latter will be referred to as *droplet density* (*D*). This droplet density may be expressed in terms of the number of droplets per foot of frontage as contrasted with the insecticide concentration *C*, which is customarily expressed as mg. per cu. m. of air. Since a characterization of an aerosol with respect to its killing power for mosquitoes requires the consideration of *D* as well as of *Ct*, it is suggested that the function *CDt* might produce a useful concept. The four factors, droplet size, time, concentration, and density, are not all variables. Fixed values of the first have been established as the optima for field work. The results in Table 2 indicate that time, which is actually determined by wind velocity, is almost a constant. Therefore, any experimentation with techniques is concerned with insecticide concentration and droplet density alone.

The optimum combination of *Ct* and *D* for forest applications was apparently approached in experiment 1, in which both the mosquito and black-fly infestations were eliminated throughout the plot. The light wind carried the aerosol slowly and the dispersal was excellent. The result is unconfirmed, but the data are worthy of consideration and comparison.

The optimum wind velocity for application over open terrain is known to be 1 to 3 m.p.h. The requirement for forest application cannot be deduced so simply as this, for in order to maintain the 1- to 3-m.p.h. air movement amongst the trees, a higher wind velocity must exist outside or above the trees. This may be referred to as the exterior wind velocity. The data shown in Table 2 may assist in the establishment of the limits of the exterior wind velocity, as measured in an area open to the full force of the wind, that will produce adequate dispersal of the aerosol within the forest. Experiment 1 indicates that an exterior wind velocity in the neighbourhood of 1 to 4 m.p.h. produced good penetration and had a good effect. However, experiments 2 and 3 make it clear that exterior velocities of 1 and 2 m.p.h. are not sufficient to produce adequate penetration of the

forest growth. In experiment 3, the aerosol remained close to the base line in ground hollows and at the tree-top level; the lower limit is therefore greater than 2 m.p.h. The other extreme, an exterior velocity of 8 m.p.h. in experiment 4, carried the aerosol rapidly and dispersed it well through the 800-yard depth of the plot and many hundreds of yards beyond. Its effect on the infestation was negligible, chiefly because of the rapidity of movement and therefore the complete reduction in the time factor. Finally, experiment 6 indicates that an exterior wind velocity of 6 m.p.h. is favourable for maximum penetration and mortality. Hence, the optimum exterior wind velocity for forest application, satisfying both demands, is in the range of 4 to 6 m.p.h.

It was stated earlier that insecticide concentration and droplet density are the variable factors that the investigator has at his disposal in the development of the most effective technique. The variation in insecticide concentration and droplet density was dependent on the selection of the droplet spectrum. The data in Table 2 indicate several significant facts in relation to these two factors. The first is the evidence that a 5 per cent DDT formulation, applied by this technique, is a satisfactory concentration and that the use of 20 per cent DDT or higher concentrations is unnecessary and uneconomical. A second observation is the significant increase in droplet density that follows a reduction of the droplet spectrum to the recommended range. This was discussed in a previous section.

It is difficult to establish the relative importance of insecticide concentration and droplet density and to separate one from the other. It is also difficult to determine their optimum levels. However, there is no evidence that the minimum droplet density has been reached. It is possible to approach the minimum droplet density level by increasing the speed of application and compensating for the subsequent reduction in the combination of Ct and D by an increase in the concentration of the insecticide in the oil solution emitted. This is also the obvious method for decreasing the time required for large-scale field operations. There is no direct evidence to support this supposition, but a personal observation of the application of this technique at Winnipeg, Manitoba, in July, 1950, suggests that it is worthy of further investigation. In this case, a 20 per cent (wt./wt.) solution of technical DDT was applied at the normal rate from a generator moving at 10 m.p.h.

CONCLUSIONS

It had been previously established that infestations of biting flies in an open, inhabited area may be controlled by the application of thermal-generated aerosols. The results of the present investigations show that ground-applied aerosols are effective in the local control of species of mosquitoes and black flies in forested areas in northern Canada. The dispersal under suitable meteorological conditions of a 5 per cent (wt./wt.) solution of technical DDT in fuel oil No. 2 or kerosene, at the rate of 8 gallons per 1,000 feet of front and with an average droplet size of 10 to 20 microns, gives effective control up to at least 800 yards in dense forest. The aerosol may be applied in such areas at any time of day providing there is within the forest an air movement of 1 to 3 m.p.h. of constant direction. A wind velocity of 4 to 6 m.p.h. above the trees is the optimum for the production of such conditions.

The technique described is effective, but not necessarily the most economical for the dispersal of aerosols. It is possible that the speed of application may be increased. However, further data are required on the physical characteristics and behaviour of aerosols, in conjunction with studies of their biological effects.

The use of a heavy aerosol generator (TIFA), under the varied and difficult conditions encountered during this investigation, leads to several observations on its practicability for large-scale control operations. The large output, flexibility, and ease of control are highly commendable features of this generator. It produces large volumes of aerosol with droplet spectrums ranging from the smallest aerosol diameters, through mists, to coarse sprays. The limited demands of a mosquito control project are thus easily met. The large volume output is an important consideration in large-scale operations, in which economy in time and material are essential. Finally, with this machine a known and constant droplet spectrum can be produced.

The large size and heavy weight of this generator require a substantial vehicle for support and transport and passable roadways or trails. These requirements are easily met in urban areas and in settled districts where such facilities exist. However, in the forest, where there are no roads the usefulness of this generator is restricted.

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PARATHION RESIDUES OF CAULIFLOWER HEADS AFTER SPRAYING[1]

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To reduce the possibility of obtaining large parathion residues on cauliflower plants at harvest, the manufacturer of this insecticide recommends (1) that the last application of spray should be applied at least 30 days before harvest or before the heads are formed, whichever date is earlier. This recommendation is not always followed because insects often appear shortly before harvest and the grower may spray in an attempt to destroy the insect population and thus maintain the quality of the cauliflower heads. The present study was undertaken to assess the extent to which cauliflower heads might be contaminated with parathion when sprays were applied later than the recommended period of application.

The external leaves of the cauliflower plant, as a rule, are tied around the head when it reaches approximately one inch in diameter. The leaves therefore would probably protect the edible portions from direct contact with spray. Untied plants at spraying would be expected to have only small heads and thus a direct application of spray to these would be diluted by growth of the head. To illustrate, a head weighing 15 grams with a residue of 0.7 p.p.m. would, if it grew to a 1-pound size at harvest, contain only 0.02 p.p.m. This decrease is only that caused by dilution from growth and does not take into consideration other losses. In view of this, direct spray contamination is probably of little concern. There is, however, the possibility of indirect parathion contamination due to absorption and translocation from sprayed leaves (5).

A single reference (2) to contamination of cauliflower by parathion stated that, at intervals of 1 to 7 days, after application of a spray containing 2 pounds of 25 per cent wettable powder per 100 gallons (U.S.) of water or a dust containing 2 per cent parathion, the residue was 0.21 p.p.m. Presumably, the residue was due to direct contamination. No information was given as to the age of the cauliflower heads when sprayed or whether they were tied at the time of spraying. The absorption and translocation of parathion from treated leaves has been investigated for some plant species. The American Cyanamid Company (1), in a summary of work from its laboratories and various state experiment stations, reported that edible portions of various types of vegetables from sprayed crops contained up to 0.025 p.p.m. parathion. Smith and Clifford (5) determined the extent to which this chemical is translocated from treated foliage to the edible portions of various plants. It was shown that the untreated internal leaves of sprayed cabbage and other crucifers, excluding cauliflower, contained from 0.13 to 0.34 p.p.m.

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MATERIALS AND PROCEDURE

A portion of a commercial field of cauliflower (variety Stokes Super Junior) near Ottawa, Ontario, was used for the investigation. The plot was 4 rows wide and 120 feet long and contained approximately 225 plants. It was divided crosswise into four equal blocks so that each row within a block contained about 14 plants. Treatments were applied at random so that each row within a block received either 1, 2, or 3 sprays, or was left untreated as a check.

The spray suspension consisted of 1 pound of 15 per cent parathion wettable powder per 100 gallons of water and was applied with a knapsack sprayer at the approximate rate of 330 gallons per acre. A canvas screen was used to reduce the possibilities of spray contamination of the adjacent non-treated plants.

Beginning on September 27, the three spray applications were made at 8- and 6-day intervals. The first, second and third sprays were applied when approximately one-quarter, two-thirds and four-fifths respectively of the plants had florets which were beginning to form. The tying of the heads was not begun until a day after the second application.

The cauliflowers were harvested when the heads reached the normal marketing stage. Because of the work entailed in extracting for chemical analyses, the four blocks were harvested a block at a time on consecutive days, starting on October 24. Intervals between the last spray and harvest were, therefore, 27, 19 and 13 days for the 1, 2 and 3 applications respectively of the first block, and 1, 2 and 3 days longer for the second, third and fourth blocks, in that order.

Sampling, Extracting and Methods

Six plants were chosen at random from each of the treated plots and four from the untreated plots. The leaves were discarded and the heads were processed as outlined below.

In order to extract both the internal and surface parathion residues from the florets—hereafter designated as internal plus external residues—they were cut into small pieces and mixed and one-half was ground to a fine pulp. Duplicate 100-gram samples were blended with 250 millilitres of benzene for 10 minutes in a Waring blendor and held overnight, and the benzene was filtered from the pulp.

Part of the mixed, unground heads from each of the treatments of the first two blocks was extracted with benzene to determine only surface parathion residues of the heads. This was done by extracting weighed quantities of approximately 500 grams of florets with 200 millilitres of benzene for 5 minutes on an end-over-end shaker.

A portion of the heads from each of the check and 3-spray treatments of the four blocks was placed in a large beaker, covered with water and boiled until tender. The water was discarded and the cooked samples of cauliflower were prepared and extracted in the same way as for the internal plus external residues of fresh cauliflower referred to above.

To obtain information on the rate of decomposition of parathion residue on the whole plant including both florets and leaves, half of the check plots remaining after the foregoing sampling was completed were sprayed at the same rate and concentration previously used on the other plots. A single

plant from each replicate was harvested immediately and 1, 3 and 6 days after spraying. Each plant was quartered and cut into small pieces. Duplicate 200-gram samples were extracted with benzene for half an hour on an end-over-end shaker. The results obtained, of course, do not give the concentration of parathion on the outer leaves but rather are average surface residues of the whole plant including leaves and florets.

Because the benzene extracts were stored at room temperature for 10 to 30 days before being analysed it was desirable to assess the possibility of parathion loss under these conditions. Analyses on known quantities of parathion dissolved in benzene cauliflower extract showed no loss during at least 59 days, the period of the experiment.

A recovery experiment was carried out to determine whether the methods of extraction and analysis for parathion were satisfactory for cauliflower tissue. Known quantities of parathion in benzene solution were allowed to permeate cauliflower through small holes drilled in the floret bases. A similar number of untreated florets from the same head were used as check samples. The cauliflower was processed and the benzene extracts were analysed by the method of Averell and Norris (4). Two such tests gave recoveries of 73 and 80 per cent.

A substance present in check samples produced a colour reaction in the chemical test. The results in this study, therefore, are reported as parts per million of apparent parathion. It was assumed that the non-parathion colour was produced to the same extent in all samples. The true parathion content is the difference between the values for the treated and check samples.

In a roughly quantitative biological test, the pomace fly, *Drosophila melanogaster* Meig., was used as a test insect to check the chemical analyses of the benzene extracts of internal plus external residues of cauliflower florets from the replicates receiving 3-spray applications. Extracts from the florets of the check plots were used as controls.

RESULTS AND DISCUSSION

The chemical analyses showed that the internal plus external residues of parathion on the edible portions of cauliflower, if present, were in such small quantities that they could not be detected. The differences between the check and treated samples were not statistically significant at the 5 per cent point.

Under the conditions of the biological test the exposure of pomace flies to a residue of 1.6 micrograms or more parathion resulted in 100 per cent mortality (*data unpublished*). Benzene extracts of 70 grams and 35 grams of cauliflower florets, designated in Table I as Series I and II respectively, from each replicate of the check and 3-spray applications treatment, were tested biologically. If these samples contained 1.6 micrograms or more parathion, which is equal to or more than 0.03 p.p.m. in the 70-gram sample and 0.06 p.p.m. in the 35-gram sample, there would be approximately 100 per cent mortality of pomace flies. Table 1 gives the percentage mortalities in the biological test for Series I and II.

The differences in mortality of pomace flies exposed to the residue from extracts of the untreated and treated cauliflower were not significant.

TABLE 1.—PERCENTAGE MORTALITIES OF *Drosophila melanogaster* EXPOSED TO BENZENE-EXTRACTED RESIDUES FROM UNTREATED AND TREATED CAULIFLOWER FLORETS

Parathion applications	Replicate	Percentage mortality	
		Series I	Series II
None	I	10	20
	II	17	23
	III	36	17
	IV	20	43
	Mean	21	26
Three	I	48	33
	II	29	36
	III	40	17
	IV	20	36
	Mean	34	31

A trace of parathion or other toxic substance may have been present; however the results suggest that the quantity of residue extracted from the treated florets was less than 0.06 p.p.m.

As would be expected from the results obtained for the internal plus external residues, the chemical analyses of surface parathion residue on florets from Replicates I and II showed that there was no measurable parathion present. Furthermore, the analyses showed that the cooked, treated cauliflower did not contain measurable quantities of parathion.

The residues on whole plants at various intervals after spray application are tabulated in Table 2 as parts per million of apparent parathion.

TABLE 2.—PARATHION RESIDUES ON WHOLE PLANTS AT VARIOUS INTERVALS AFTER SPRAYING

	Replicate	Days after spraying			
		0	1	3	6
		Apparent p.p.m. parathion			
Check	I	0.5	0.4	0.6	0.8
	II	0.4	0.6	0.6	1.3
	III	0.2	0.7	1.0	1.0
	IV	0.5	0.6	0.5	1.4
	Mean	0.4	0.6	0.7	1.1
Treated	I	1.1	1.1	0.4	1.4
	II	1.6	1.1	1.2	1.5
	III	1.0	1.1	1.3	0.7
	IV	0.7	1.3	1.8	—*
	Mean	1.1	1.2	1.2	1.2

* Sample lost.

A single application of spray resulted in a measurable quantity of approximately 0.7 p.p.m. parathion residue for the whole plant. This was present until about the third day, when it apparently disappeared. On the sixth day, however, the values for the check samples were very high. This may have been due to contamination by spray drift but it is more likely that severe frosts and snow a few days before the last harvest affected the plants in such a way as to cause an increase in benzene-extractable, natural colour-producing substances. At the same time, the snow and moisture as well as natural volatilization and decomposition probably lowered the true parathion residue.

SUMMARY AND CONCLUSIONS

The chemical and biological analyses showed that cauliflower heads taken from plants sprayed up to three times at approximately weekly intervals with 1 pound of 15 per cent parathion per 100 gallons of water showed no residues, either absorbed within or on the surfaces of the florets, which were significantly greater than those apparent on unsprayed checks. It was possible that there were traces of the insecticide present in or on florets. The residue, however, was well below the unofficial tolerance of 1.5 p.p.m. The results of this investigation suggest that there does not appear to be any danger of obtaining toxic quantities of parathion in or on the edible parts of cauliflower if this insecticide is applied at the rate recommended (3) for other vegetables.

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EXPERIMENTAL TRANSFERS OF THE BULB NEMATODE OF IRIS, OF NARCISSUS, AND OF HYACINTH TO POTATO[1]

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INTRODUCTION

The distribution of the potato rot nematode, *Ditylenchus destructor* Thorne, in North America is, according to present information, rather limited, for the species has been reported only from Prince Edward Island in Canada and from Idaho in the United States. However, recent evidence and research suggest that the bulb nematode of iris, at present classified as a form of *D. dipsaci*, may be identical with it, and, if this should prove to be true, the distribution of *D. destructor* will be greatly widened, for the bulb nematode of iris is widespread in British Columbia and Washington, and probably occurs wherever irises are grown commercially. It is widespread in the iris plantings of Holland and Great Britain.

The classification of the iris nematode as a form of *D. dipsaci* has been in doubt for a considerable time. Goodey (1), in 1950, stated that this doubt has existed in Great Britain for 25 years. In 1945, Van Slogteren (7), in referring to his transfer studies, stated that the behaviour of the iris nematode convinced him that it is not a form of *D. dipsaci*.

Specimens of iris nematode collected in British Columbia from iris bulbs were taken by one of the authors to the Station of the Division of Nematology, United States Department of Agriculture, Salt Lake City, Utah, and compared with type specimens of *D. destructor*. No morphological distinctions were found. Goodey (2) examined nematodes from diseased iris bulbs in England and found that they had rounded tail tips and six incissures in each lateral field; characters by which *D. destructor* is distinguished from *D. dipsaci*. He conducted cross inoculation experiments and, as a result of these and of his examination of the nematodes, concluded that *D. destructor* is the nematode that causes the eelworm disease of bulbous iris. On this continent, no decision regarding the reclassification of the iris nematode has yet been made, and no publication describing the iris nematode as a new species or as a form of *D. destructor* has appeared in the literature. However, quarantine officers and producers of potatoes and irises are very much interested in the host-parasite relationship of potato to the iris nematode, and of iris to *D. destructor* from potato.

Conditions affecting transfers of nematodes

As plant parasitic nematodes are obligate parasites, difficulties with respect to inoculum, and its source and condition, have often been responsible for failure of many inoculations to effect transfers. Investigators cannot assume that the application of plant material infected with nema-

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todes will ensure the entry of the nematodes into a susceptible host, for one or more of the factors discussed below may influence the results of the inoculation.

(a) If the source of inoculum is not a highly susceptible host, the nematode population may be very small. For example, the bulb tissue of iris Supreme carries about 60 times as many nematodes as that of iris Imperator (3). A worker using iris Supreme as a source of inoculum has, therefore, a much greater chance of obtaining a transfer than one using iris Imperator.

(b) Plant parasitic nematodes tend to become dormant in the presence of many substances, including the products of plant decay and of bacterium or fungus metabolism.

(c) Such nematodes cannot be maintained long in an active condition if the depth of the substrate is too great or the air supply inadequate.

(d) Nematodes in a liquid suspension do not have as good support for forcing an entry into a plant as if they were in soil or on a solid medium.

(e) A specific host-parasitic relationship should not be considered proven until it is established that the parasite has completed its life cycle in the host. Except when the inoculum used consists of nematodes in the larval stages, it cannot be assumed that, after transfers have been made, the presence of eggs, larvae, and adults in the tissue of plants is evidence of a susceptible host. For instance, gravid females, 3 weeks after removal from host tissue to water, were observed to be still producing eggs. It would appear quite possible, therefore, that adults, eggs, and larvae may be present in a plant and yet the plant not be a favourable host.

When the source of the inoculum and the plant to which the transfer is to be made are both highly susceptible varieties, positive results usually follow, provided that none of the factors mentioned above seriously intervenes. A knowledge of the susceptibility of a plant species and its varieties is, therefore, useful to students of nematode forms. The susceptibility of iris varieties to the iris nematode originating in iris Supreme was studied by one of the authors (4). The varieties studied were classified as follows:

Highly Susceptible: Supreme, White Excelsior, Bloemaard, Thunderbolt, King of the Blues, P. de Ridder.

Moderately Susceptible: Hart Nibbrig, Van Everdingen, Cornelia, Prince Albert.

Slightly Susceptible: Wedgewood, Imperator, Yellow Queen, Poggenbeek, Queen Wilhelmina, Cajanus, Count of Nassau, Louis.

Resistant: La Renaissance, King of the Whites.

Similarly, a study was made of the varietal susceptibility of potatoes to a species of *Ditylenchus* from *Tropaeolum polyphyllum* (5). The varieties studied were classified as follows:

Highly Susceptible: Katahdin, Green Mountain, Warba.

Moderately Susceptible: White Rose, Houma, Early Epicure, Irish Cobbler.

Slightly Susceptible: Raleigh, Early Rose, Sutton's Reliance, Beauty of Hebron.

Resistant: Netted Gem.

EXPERIMENTAL

Some preliminary studies were undertaken with a view to the experimental transfer of the iris, of the narcissus, and of the hyacinth bulb nematode to potato, care being taken to obviate failure arising out of any of the factors referred to above.

Source of inoculum

The narcissus form presents the least difficulty with respect to source of inoculum, for, when the nematodes move down the infected scale of a bulb into the base, they assemble in colonies of almost pure pre-adults. These colonies become dry and dormant. As they have the appearance of bits of wool, they have been described as "eelworm wool". Eelworm wool of the narcissus form may be collected and stored, and the worms will remain viable for at least two years. When required for transfer experiments, wool of the narcissus form may be suspended in water, and, as it carries little or no contamination, the nematodes will continue active in the water until death ensues from exhaustion.

The iris and hyacinth bulb nematodes, on the other hand, do not assemble in such colonies, and so, to obtain inoculum of them, infected plant tissue must be teased out in water. The iris form used in the present study was obtained from English iris, *I. xiphoides*, variety King of the Blues. This variety has been classified by the authors as highly susceptible, as infected bulbs carry large populations. When collected, the plants were near flowering, and it was found that some of the nematodes had passed from the originally infected scales of the parent to current season growth, entering the leaf sheaths and some of the stems. Denser nematode populations, with good proportions of larvae and eggs, were found in the leaf sheaths than in the bulb scales. Furthermore, nematode suspensions obtained by teasing infected leaf sheath tissue in water were relatively clear. In contrast, nematode suspensions, obtained by teasing infected bulb scales, carried an abundance of starch particles and other contaminants, and a quick change of the nematodes from this substrate to another was difficult. The leaf sheaths were, therefore, a better source of the iris nematode than the bulb scales.

The hyacinth nematode was obtained from plants in full bloom. The leaves showed typical spikkel symptoms. However, quite often the section where the conspicuous symptoms were located yielded only a few nematodes. Population distribution of five half-inch sections of a leaf cut consecutively from the base to a little beyond the location of spikkels gave counts of 136, 63, 11, 23, and 16 nematodes respectively. The population had migrated to the base and the basal section was the best source of nematodes, notwithstanding the presence of the symptoms elsewhere.

Longevity of nematodes in water suspension

It is known that in tap-water the narcissus nematode will remain alive for over a month, but the viability of the iris and of the hyacinth nematode is less well known. The data presented in Table 1 are counts made on 14-day-old suspensions of these two nematodes. As shown in Table 1, the young larvae of the hyacinth nematode were weaker than those of the iris nematode, and most of them were dead within 14 days.

TABLE 1.—VIABILITY OF THE IRIS AND THE HYACINTH NEMATODE AFTER 14 DAYS IN WATER

Class of nematodes	Iris nematode			Hyacinth nematode		
	Number motile	Number non-motile	Percentage survival	Number motile	Number non-motile	Percentage survival
Adult	39	0	100	12	5	74
Pre-adult	6	0	100	5	1	83
3rd stage larvae	5	3	62	2	2	50
1st and 2nd stage larvae	49	4	92	7	136	5

Preparation of inoculum

Linford (6) originated a method of separating root-knot nematode larvae from attendant plant debris. The suspension of nematodes and debris is poured into a small folded paper filter in a stemless funnel and is washed down into the point. The funnel is then lowered until the tip of the paper makes contact with water in a small container and the funnel is capped with a Petri dish cover to prevent desiccation. Within from 12 to 48 hours most of the nematodes wriggle through the paper into the water. The method depends on the motility of the nematodes.

The nematodes obtained from plants by teasing infected plant tissue in water will be in a liquid with suspended particles of fibre and starch, and also plant juices and other matter that may be in solution. In such an environment, nematodes soon lose their motility. It is necessary, therefore, to effect a quick change of the nematodes from such a substrate to another one in which rapid inactivation does not take place. In the present experiments, this was done by removing the coarse plant material from the suspension and then passing it through a one-half-inch plug of cotton wool in the stem of a funnel. Two rinsings with tap water followed. The cotton plug was then placed in the water over a filter paper, the central portion of which was submerged in a Syracuse watch glass partially filled with tap water. The nematodes worked their way through the paper to the water below. When the time allowed for their passage was less than 24 hours, the nematodes that passed through the paper were predominantly 1st and 2nd stage larvae, but, when more time was allowed (for example, 48 hours), all stages passed through all the brands of filter paper tested, including Whatman Nos. 1, 2, 5, 40, and 50. Adults that passed through the filter paper were, soon afterwards, producing eggs. These eggs hatched in 6 days. By repeatedly screening out the eggs, it was established that egg-laying was not completed three weeks after gravid females were separated from the host. In one instance, a female lost the ability to expel her eggs and twelve larvae hatched and were entombed in her body. The capacity of a gravid female to produce that many young under the circumstances shows how easy it is to misinterpret a host-parasite relationship from transfers effected with unsorted inoculum.

Inoculation method for transfers to potato

Nematodes that were freed of plant residue by filtration through cotton wool and by passage through filter paper into tap water were assembled in shallow dishes. By means of a pipette, a portion of the suspension was added to 5 cc. of water in a Syracuse dish and examined under the low power of a dissecting microscope in order to assure uniformity of size in the population. All off-size individuals were removed. Suspensions thus prepared were adjusted to 20 worms to each microscopic field. A pad of cotton wool was then placed in the dish, and the pad took up the nematodes. A potato tuber was then placed on the pad, and contact between the potato and the inoculum was maintained for 21 or 35 days, as the case might be. The cultures were kept in a moist chamber and, as occasion required, the pads were moistened by the addition of water. In one series, the skin of the tubers was left intact; in another series, it was punctured by a needle. The cotton pad gives the nematodes a support that is lacking in a liquid medium. It also increases the surface of the medium, thus assuring an adequate air supply and continued activity of the population. Moreover, advance information on the result of inoculation is quickly available. For instance, in the experiment with the narcissus nematode, evidence of rapid entry into the pricked potato was obtained by placing the cotton pad that was under a pricked potato and that under an unpricked potato each in a different dish of water. The populations, which were equal at the start, showed 12 hours later a ratio of almost 10-to-1 in favour of the unpricked potato. That is to say, entry into the pricked tuber was much more rapid than into the unpricked tuber.

Results

In all cases in which the skin of the potato was punctured, positive transfers of the iris, the narcissus, and the hyacinth nematode to potato tubers were effected. However, when the tubers were not punctured, the narcissus nematode was better able than the iris nematode to effect an entry, and the hyacinth nematode did not enter at all.

Inoculations with the iris nematode were also made with unsorted nematodes in one series, and with 1st and 2nd stage larvae in another series. In all cases, the nematodes recovered from the potato included adults, gravid females, young, and eggs. The nematodes were able to complete their life cycles. There is therefore a true host-parasite relationship between the iris nematode and the potato.

The inoculations with the hyacinth nematode were made with 1st and 2nd stage larvae, and those of the narcissus nematode with pre-adults. In both cases, the nematodes transferred, fed, grew, and reached sexual maturity, but, at the end of 35 days, there was still no evidence of gravid females or eggs. The life cycle was not completed, so it cannot be concluded that there is a true host-parasite relationship between either the hyacinth or the narcissus nematode and the potato. Apparently the potato acted only as a reservoir host for these two nematodes. The data are presented in Table 2.

TABLE 2.—NUMBER OF NEMATODES RECOVERED FROM POTATO TUBERS INOCULATED WITH NEMATODES FROM IRIS, FROM NARCISSUS, AND FROM HYACINTH

Form	Kind of inoculum	Tuber treatment	Days after inoculation	Number and kind of nematodes						
				Gravid females	Females	Males	Pre-adult	3rd larval	1st and 2nd larval	Eggs
Iris	Mixed adult and young	Pricked	21	1	0	1	1	2	4	4
"	"	"	21	1	1	4	3	0	5	2
"	"	"	21	0	4	0	0	0	4	6
"	"	"	21	1	2	2	2	4	3	2
"	"	"	21	3	2	0	0	0	2	2
"	"	"	21	4	2	2	1	1	3	3
"	1st and 2nd larval stages	"	35	3	5	5	6	13	57	4
"	"	"	35	1	6	4	4	10	32	3
"	"	"	35	2	4	4	4	6	31	5
"	"	"	35	2	4	2	3	4	25	3
"	"	"	35	3	3	4	4	5	26	6
"	Mixed adult and young	Non-pricked	21	0	0	0	1	0	2	0
"	"	"	21	0	0	0	0	1	3	0
"	1st and 2nd larval stages	"	35	0	0	0	0	1	1	0
"	"	"	35	0	0	0	0	2	4	0
"	"	"	35	0	0	0	0	0	0	0
Hyacinth	"	Pricked	35	0	1	0	1	1	6	0
"	"	"	35	0	1	0	1	2	4	0
"	"	"	35	0	2	0	1	2	3	0
"	"	Non-pricked	35	0	0	0	0	0	0	0
"	"	"	35	0	0	0	0	0	0	0
"	"	"	35	0	0	0	0	0	0	0
Narcissus	Pre-adult	Pricked	35	0	12	5	23	0	0	0
"	"	"	35	0	3	1	28	0	0	0
"	"	"	35	0	6	3	24	0	0	0
"	"	Non-pricked	35	0	1	0	8	0	0	0
"	"	"	35	0	3	1	13	0	0	0
"	"	"	35	0	2	1	12	0	0	0

SUMMARY

The common and widely distributed bulb nematode of iris is indistinguishable morphologically from the potato rot nematode *Ditylenchus destructor* Thorne. First- and second-stage larvae of the iris nematode will enter, and complete their life cycle, in potato tubers. Larvae of the narcissus bulb nematode will also enter potato tubers, but they fail to complete their life cycle in them. Larvae of the hyacinth bulb nematode enter only potato tubers that are injured, and they likewise fail to complete their life cycle in them. The presence of gravid females, eggs, and larvae in a host is not conclusive evidence of true nematode parasitism, for gravid females will lay eggs in tap-water and these eggs will hatch there over a period of 21 days. Transfers to potatoes are satisfactorily effected when wads of wet cotton wool containing active larvae of a single stage are placed on the tubers. In the preparation of the inoculum for the wads, the original macerated plant material containing the nematodes is washed to remove substances that inactivate the nematodes, then larvae of a single stage are isolated from the original mixed population by passage through filter papers.

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THE EFFECT OF SMALL ADDITIONS OF ELEMENTAL SULPHUR ON THE AVAILABILITY OF PHOSPHATE FERTILIZERS[1]

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Field trials in 1948 in Saskatchewan indicated the relatively low availability of dicalcium phosphate as a phosphate fertilizer. The suggestion has been made that this may be due to a more rapid alteration to $Ca_3(PO_4)_2$ or some related product, or to a low solubility, both being due to the neutral or alkaline reaction and high calcium levels of the soils studied (2).

If this hypothesis is correct, the incorporation of a little elemental sulphur into the fertilizer might, by virtue of microbiological oxidation in the soil, induce a lower pH in the environment of the fertilizer granule, and hence hinder "fixation" and thereby favour higher solubilities.

A preliminary experiment (3) in the greenhouse in 1947–48 in which 5, 10 and 15 per cent of flowers of sulphur was added to mono-ammonium phosphate showed that significant yield increases of grain resulted from adding the sulphur. There was no deficiency of sulphur in the soil which was from the surface layer of a solonetsous chestnut soil on Lacustrine material with a reaction slightly below neutral (pH. 6.7).

EXPERIMENTAL

Greenhouse Experiment 1948–49.

On the basis of the results obtained in the 1947–48 experiment, a second greenhouse experiment was set up in 1948–49 using P^{32} as a radioactive tracer to follow the uptake of phosphorus more closely. Thatcher wheat was used as the test crop, with 4 plants per pot, each of which contained 3200 gm. of soil from the surface layer of a thick black soil on glacial till. The reaction of the soil was neutral to slightly alkaline (pH 7.4). Two phosphates, mono-ammonium phosphate (11–48–0) and dicalcium phosphate-nitrate (8–30–0) were used at a rate equivalent to 24 pounds of P_2O_5 per acre. With each phosphate four levels of sulphur were used, equivalent to 0, 0.5, 1, and 5 per cent by weight of the mono-ammonium phosphate.

The labelled fertilizers were prepared as follows:

Mono-ammonium phosphate (11–48–0)—The required amount of inactive material was dissolved in water, P^{32} added as $P^{32}O_4^{\equiv}$, and the whole evaporated to dryness at less than 45° C. The amount required for each sulphur treatment was weighed out, redissolved, and the appropriate amount of flowers of sulphur added. Stirring during the subsequent evaporation gave some control over particle size and ensured thorough mixing of the sulphur throughout the material.

Dicalcium phosphate nitrate (8–30–0)—In the greenhouse trials, approximately one-third of the required amount of inactive material was dissolved in 25 per cent acetic acid at 50° C., the P^{32} added as $P^{32}O_4^{\equiv}$, and

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evaporated to dryness at less than 33° C. This active material was then ground and mixed by shaking with the remaining two-thirds of the material containing the required amount of sulphur. Where larger amounts of this material had to be prepared as for field tests, about one-tenth was dissolved to avoid evaporation of large quantities of solution. Active phosphorus was added as above. To the homogeneous mixture enough water was added to make a thick paste. The mixture was forced through a 3-mm. sieve to pellet the material comparable in size to the mono-ammonium phosphate, and dried.

The seed and fertilizer were placed in a layer 2 inches below the surface to approximate field methods of placement of the seed. Six replicates of each treatment were used. The plants were harvested at the heading stage. At harvest, the portion of the plants above the crown were dried, weighed and analysed for total phosphorus using the wet-ashing method of Brenner and Harris (1) and the molybdenum blue method with hydrazine as the reducing agent (4). P^{32} was estimated by counting $Mg_2P_2O_7$ as outlined by Spinks and Barber (5).

The results are given in Table I.

TABLE 1.—THE EFFECT OF ELEMENTAL SULPHUR ON FERTILIZER PHOSPHATE UTILIZATION IN THE GREENHOUSE

Fertilizer	Per cent S added	Plant Wt. lb./acre	Total Uptake of P_2O_5 lb./acre	Soil P_2O_5 in plant lb./acre	Fert. P_2O_5 in plant lb./acre	% Fertilizer P used
Dicalcium phosphate + Calcium nitrate 24 lb. P_2O_5/acre	0	1426	8.4	6.0	2.3	9.6
	0.5	1675	9.4	6.7	2.7	11.4
	1.0	1658	9.7	6.2	3.5	14.7
	5.0	1620	10.1	5.3	4.8	19.9
Ammonium dihydrogen phosphate 24 lb. P_2O_5/acre	0	1608	11.3	7.4	3.9	16.3
	0.5	1782	11.6	7.4	4.2	17.7
	1.0	1771	11.3	7.1	4.2	17.3
	5.0	1600	10.9	5.7	5.0	20.6
L.S.D. (.05)		116	0.7	0.8	0.5	1.9

The data show that the percentage of fertilizer phosphorus taken up by wheat from the dicalcium phosphate-nitrate increased directly with the amount of incorporated sulphur, and that the 5 per cent sulphur addition doubled the uptake of phosphorus in comparison to the treatment with no sulphur. The treatment with 5 per cent sulphur was sufficiently effective in increasing the availability of dicalcium phosphate-nitrate to make it at least as available as untreated mono-ammonium phosphate.

Even with mono-ammonium phosphate, the highest rate of sulphur addition was effective in significantly increasing uptake and availability.

Field Experiments, 1949

In 1949, an experiment was set out at two locations, one at Watson on a calcareous black soil on glacial till with an alkaline reaction (pH 8.4) and the other at Birch Hills on a thick black soil on lacustrine material (pH 7.2). Phosphate was applied at 24 pounds P_2O_5 per acre as mono-ammonium phosphate, mono-calcium phosphate and dicalcium phosphate-nitrate, with sulphur additions at the rate of 0, 1 and 5 per cent of the weight of mono-ammonium phosphate used. P^{32} was added as a tracer to detect any changes in fertilizer uptake due to the sulphur treatment. Thatcher wheat was used at both locations.

The data obtained from yields and by analysis of the above-ground parts as given in Table 2, indicate no effects of any sort due to the sulphur additions. There are no differences due to the sulphur treatment in the amounts of fertilizer phosphorus taken up for any of the three materials. The data in no way confirm the previous winter's greenhouse experiment on the effectiveness of sulphur in promoting the uptake of phosphorus.

TABLE 2.—THE EFFECT OF ELEMENTAL SULPHUR ON FERTILIZER PHOSPHATE UTILIZATION IN FIELD TRIALS, 1949*

(Thatcher wheat harvested at maturity)

Fertilizer used at 24 lb. P_2O_5/acre	Per cent S added	Plant wt. gm.	Grain yield gm.	Fert. P in plant mgm.	Soil P in plant mgm.	% Fert. P used
Birch Hills						
Mono-ammonium phosphate	0	220	96	110	227	33.6
	1	199	86	102	193	31.2
	5	213	92	102	200	31.0
Mono-calcium phosphate	0	210	93	75	240	23.0
	1	189	82	71	224	21.7
	5	205	91	80	235	24.6
Dicalcium phosphate-nitrate	0	162	70	16	247	4.8
	1	160	70	19	239	5.7
	5	178	76	17	251	5.1
No Fertilizer	0	149	65	—	239	—
L.S.D. (5% level)		27	11	9	35	2.8
Watson						
Mono-ammonium phosphate	0	256	95	79	269	24.0
	1	252	96	78	261	23.9
	5	260	100	78	277	23.9
Mono-calcium phosphate	0	261	98	66	209	20.2
	1	200	73	16	276	4.7
Dicalcium phosphate-nitrate	0	224	81	16	308	5.0
	1	267	102	70	314	21.3
	5	242	91	65	277	19.9
	5	208	75	15	278	4.7
No Fertilizer	0	212	76	—	287	—
L.S.D. (5% level)		30	11	14	N.S.	4.4

* Results based on six replicates of a 6′ row in a randomized block arrangement.

The failure of the 1949 field experiments to confirm the greenhouse data suggested that sulphur oxidation had been very slow under field conditions. The fact that lower temperatures would prevail in the field than in the greenhouse could be a factor in reducing the rate of oxidation. A second factor is that there is no certainty that an active population of S-oxidizing organisms exists in all soils at all times and so there might be a period of delay before oxidation would proceed actively.

The 1949 data confirmed work previously cited (2), further demonstrating the greater efficiency of mono-ammonium phosphate over mono- and dicalcium phosphates for cereals on neutral to alkaline soils in Saskatchewan.

Field Experiments 1950

A second field experiment was designed to find out if treating the S-containing fertilizers with a suspension containing S-oxidizing organisms would make the S-treatment more effective, the presumption being that the indigenous S-oxidizing organisms were too few in number or too inactive to be effective.

The treatments which are outlined in Table 3 consisted of:

(1) Control
(2) 4 pounds of sulphur per acre, treated as indicated below
(3) Mono-ammonium phosphate (11–48–0)
(4) Dicalcium phosphate-nitrate (8–30–0)
(5) Dicalcium phosphate-nitrate (8–30–0) plus 5 per cent sulphur
(6) Dicalcium phosphate nitrate (8–30–0) plus 5 per cent sulphur treated as described below.

The fertilizers were obtained from the United States Department of Agriculture.* The materials were granulated, and where required, mixed with flowers of sulphur as described previously.

The sulphur and 8–30–0 in (2) and (6) were treated with a suspension from a mixture of soil, manure, peat and sulphur, which was actively oxidizing sulphur as indicated by the acid reaction. The suspension was applied to the granules of 8–30–0 plus S fertilizer. This crude inoculum did not contribute any significant amount of material other than microbiological organisms.

Thatcher wheat was used as the test crop and was harvested after heading.

The trials were located at Birch Hills, a thick black soil on lacustrine material of near neutral reaction and at Waitville on a slightly acid grey-wooded podzolic soil on glacial till. Both fields had been fallowed the previous year.

The data obtained by analysis of the above-ground parts are shown in Table 3 giving the data for each location, and the mean results for both.

In agreement with the results of the field experiments of 1949, the addition of sulphur to 8–30–0 alone does not increase its availability. If S-oxidizing bacteria are added, however, the oxidation of sulphur causes a significantly increased uptake of fertilizer phosphate, presumably as a result

* Through the courtesy of L. T. Alexander, of the U.S.D.A., Beltsville, Maryland.

TABLE 3.—EFFECT OF ELEMENTAL SULPHUR ON FERTILIZER PHOSPHATE UTILIZATION IN THE FIELD, 1950*

P_2O_5 = 24 lb./acre for fertilizer treatments

Fertilizer treatment	Plant wt. lb./acre in hundreds	Phosphate taken up by plant			
		Total P_2O_5 lb./acre	Soil P_2O_5 lb./acre	Fert. P_2O_5 lb./acre	% Fert. P used
Waitville, 1950 (August 3)					
Check	25.7	10.6	10.6	—	—
Check + S treated	28.1	10.6	10.6	—	—
11–48–0	37.1	11.9	6.0	6.0	24.9
8–30–0	26.4	8.9	8.0	1.0	4.1
8–30–0 + 5% S	31.5	10.8	9.8	1.0	4.1
8–30–0 + 5% S treated	30.0	9.9	8.3	1.7	6.9
L.S.D. (0.05)	5.9	2.1	2.1	0.4	
Birch Hills, 1950 (August 8)					
Check	44.9	21.0	21.0	—	—
Check + S treated	43.2	20.1	20.1	—	—
11–48–0	54.0	23.8	18.1	5.8	23.9
8–30–0	49.1	22.4	21.2	1.1	4.5
8–30–0 + 5% S	50.7	23.1	22.0	1.1	4.6
8–30–0 + 5% S treated	51.4	23.2	20.8	2.3	9.8
L.S.D. (0.05)	7.2	3.4	3.3	0.7	
Mean of both locations					
Check	35.4	15.7	15.7	—	
Check + S treated	35.5	15.3	15.3	—	
11–48–0	45.8	17.8	12.0	5.86	
8–30–0	37.8	15.6	14.6	1.03	
8–30–0 + 5% S	41.1	16.9	15.9	1.03	
8–30–0 + 5% S treated	41.0	16.4	14.4	1.98	
L.S.D. (0.05)	4.5	N.S.	1.8	0.40	

* Results based on 6 replicates of a 12-ft. row in a randomized block arrangement.

of the maintenance of a reduced pH in the environment of the fertilizer granule, and consequently, either a reduced rate of fixation or an increased solubility.

The increased availability of phosphate in the 8–30–0 + S treated with inoculum over the untreated material has shown no increase in yield of plant material in these data, nor any significant change in uptake of phosphorus from the soil.

Although it has been demonstrated that the action of S-oxidizing bacteria on elemental S in the fertilizer doubled the availability of di-calcium phosphate-nitrate, it can be calculated that even this more effective di-calcium phosphate nitrate is only about 30 per cent as effective as mono-ammonium phosphate (11–48–0) in supplying phosphorus to cereals under the conditions of the experiment. While it is conceivable that variations in the form and amount of added sulphur might give somewhat different results, it seems probable that the most significant barrier to the successful use of sulphur as an amendment in such materials as di-calcium phosphate is probably due to the low activity of the indigenous S-oxidizing microbes of the soil under the conditions of the field tests.

The 1950 experiments both explain and confirm the previous data. In agreement with the 1949 field data, the addition of sulphur alone to dicalcium phosphate-nitrate was without effect, while in the presence of S-oxidizing organisms, a significant increase in fertilizer phosphate uptake was obtained, as in the greenhouse experiment. The greenhouse data are partly explainable as being the result of increased rates of sulphur oxidation by the natural flora promoted by the higher soil temperatures and better moisture conditions prevailing in the greenhouse.

SUMMARY

Greenhouse experiments have shown that a relatively inefficient phosphate carrier for neutral to alkaline soils such as 8–30–0 dicalcium phosphate-nitrate could be greatly improved as a supplier of phosphate to cereals by the addition of small amounts of elemental sulphur. The uptake of phosphorus from 8–30–0 plus sulphur as measured by the tracer technique was shown to be equal to that obtained from the more efficient monoammonium phosphate (11–48–0) fertilizer.

In field experiments in 1949 sulphur additions failed to show any measurable effect in increasing the uptake of phosphorus from the 8–30–0 fertilizer.

The field experiments were repeated in 1950 with the addition that the sulphur was treated with an infusion from a soil, manure and peat mixture to which sulphur had been added previously. A significant increase in uptake of phosphorus occurred where 8–30–0 plus treated sulphur was used, but not where untreated sulphur was added. However, in the field tests 8–30–0 was only about one-third as efficient as 11–48–0 in supplying phosphate to cereals. Addition of treated sulphur to the fertilizer was, therefore, considerably less efficient in field test than was the addition of sulphur without treatment in greenhouse trials.

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EFFECT OF 2,4-D ON QUALITIES OF WESTERN CANADIAN WHEAT, BARLEY, AND OATS[1]

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INTRODUCTION

Control of weeds in standing grain by use of 2,4-D (2,4-dichlorophenoxyacetic acid) is now established farm practice in Western Canada. In 1950, approximately 13 million acres of small grain crops were treated with formulations of this chemical.

Although general use of 2,4-D on grain crops is a comparatively recent practice, Heal and Thompson (3) show that a large volume of work has been published on this herbicide for controlling weeds injurious to agronomic crops. Many aspects of the subject have been investigated, but yield per acre has been the chief criterion for judging efficiency of treatments. Scant attention has been paid to the qualities of grain crops that have been sprayed or dusted during the growing period. In the recent and extensive studies made in Denmark on the spraying of wheat, oats, and barley with 2,4-D and 4K-2M, Anderson and Hermansen (1) confine their data to yield, test weight, germination, and abnormalities in the straw, ears, and kernels.

The few papers that do refer to the effect of 2,4-D on grain quality deal mostly with wheat, and then principally with protein content and bushel weight. Erickson, Seely, and Klages (2) report that applications of 2,4-D heavy enough to kill sensitive weeds increased the protein content of wheat, and that the increase did not appear to depend on weed competition, variety, stage of growth prior to heading, or whether the land was dry or irrigated. Helgeson (4) also found a general increase in wheat protein from 2,4-D treatments, and that variations in protein of varieties differed with the kind of formulation used and the growth stage at time of application. Mitchell and Linder (7) found that the increase in protein content of 2,4-D treated Pilot wheat was often associated with a corresponding decrease in bushel weight. Klingman (6) observed a reduction of about 5 pounds in the bushel weight of Pilot wheat with a minimum concentration of 2,4-D applied at the boot stage of kernel development.

Helgeson, Blanchard, and Sibbitt (5), studied the effect on the milling and baking qualities of several wheat varieties of standard dosages of the ester, amine, and sodium salt formulations applied at different growth stages. Bushel weight of Mida was relatively constant for all treatments, but there was a significant rise in protein content for the amine and ester treatments at the tiller stage and for all formulations at the boot and bloom stage. The amine and the ester caused a general reduction in loaf volume at the boot and soft-dough stages and significant increases at the tiller stage with the ester and amine. For varieties treated only at the

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tiller stage there was no significant reduction in bushel weight, but there were significant increases in protein content for Thatcher, Pilot, Regent, and Mindum with the ester, and for Mida, Regent, and Thatcher with the amine; the sodium salt and the amine caused a significant reduction in the protein content of Carleton. There was, however, no significant reduction in loaf volume. In a similar study, Sibbitt and Harris (10) concluded that applications of the acetate, the triethyl amine, and the butyl ester of 2,4-dichlorophenoxyacetic acid to wheat plants at different growth stages did not impair milling and baking quality. With some treatments there was some evidence that baking quality was improved. For hard red winter wheat representing different dates, types, and dosages of 2,4-D application, Shellenberger, Phillips, Johnson, and Miller (9) report that there was no effect on mineral content, milling quality, or baking quality. So far as the authors of the present paper know, there is no previous reference in the literature to the qualities of 2,4-D treated barley and oats.

In 1948 and 1949, studies were made by the Department of Plant Science, University of Manitoba, to obtain information on yield per acre and other agronomic characters of various cereal grains treated at different stages of plant growth with different formulations and dosages of 2,4-D. Data obtained for barley and wheat have been published by Olson, Zalik, Breakey, and Brown (8). To supplement these studies, several sets of hard red spring wheat, amber durum wheat, barley, and oats were submitted to the Grain Research Laboratory for quality tests. This paper deals with results of the quality tests made on these samples.

The paper is presented in two parts. Part I deals with grade, bushel weight, and protein content of different varieties of bread wheat, durum wheat, barley, and oats, and Part II with the milling, baking, and other qualities of a series of Redman wheats and with the barley and malt properties of sets of barley. Data presented in Part I are for samples treated with three 2,4-D formulations in 1948 and with two formulations in 1949. Data presented in Part II for wheat are for 1949 only, but data for both 1948 and 1949 are given for barley. Additional years' data for this part of the study would obviously have been desirable, but those that are presented and discussed should be a useful guide in future studies of this kind.

PART I—GRADE, BUSHEL WEIGHT, AND PROTEIN CONTENT

In considering the data presented in this part of the paper, it should be borne in mind that grade is a good index of kernel soundness, that bushel weight is a measure of kernel size and plumpness, and that protein content is a useful measure of quality. The data for 1948 and 1949 are discussed separately.

1948 Samples

The 1948 study involved spraying quadruplicate rod rows of Thatcher and Redman hard red spring wheat, Stewart amber durum wheat, Vantage and Montcalm barley, and Ajax and Exeter oats, with three dosages of each of three formulations of 2,4-D on two different dates at Winnipeg. Formulations used were the sodium salt, the amine, and the ester. Dosages were 0, 4, 8, and 16 ounces per acre of the acid equivalent, and dates of

application were May 29 and July 5. On May 29, all plants were 4 to 6 inches tall. On July 5, growth stage was about 10 days after heading for Thatcher and Redman, 1 to 2 days before heading for Stewart, 5 to 6 days after heading for Vantage and Montcalm, 3 days before heading for Exeter, and 5 days after heading for Ajax. In discussing the data, the first application is referred to as the blade stage and the second application as the head stage.

Grades were assigned by the Winnipeg Inspection Office of the Board of Grain Commissioners, and bushel weight and protein determinations were made by the Laboratory. Protein content (N × 5.7 for wheat, N × 6.25 for coarse grains, 13.5 per cent moisture basis) was determined in quadruplicate on samples from each quadruplicate rod row, and significant differences were calculated. For grade and bushel weight, it was necessary to composite quadruplicate samples to provide larger ones for these determinations. Even after compositing, several samples were too small to determine bushel weight with a pint measure. When this occurred, a gill measure was used for all samples in the same group so as to obtain comparable results. Since bushel weight was being studied separately, grades were assigned on the basis of kernel appearance and soundness. For most samples, bushel weights were equal to or higher than the minimum required for the grade. When, however, the bushel weight of a particular

TABLE 1.—BUSHEL WEIGHT DATA 1948 CROP. DIFFERENCES BETWEEN 2,4-D TREATED AND CONTROL SAMPLES

	Sodium salt				Amine				Ester			
Dosage, oz.	0	4	8	16	0	4	8	16	0	4	8	16
Thatcher wheat—												
Blade	62.2	0.0	0.8	−0.2	62.2	−0.2	0.3	0.8	61.8	1.0	0.4	1.0
Head	62.8	−0.3	0.2	0.4	62.5	−0.5	0.0	−0.3	62.2	0.3	−1.7	−1.0
Redman wheat—												
Blade	63.5	0.0	−0.3	−0.3	63.2	0.0	−0.4	−1.0	63.2	−0.2	−0.4	−0.7
Head	63.5	−0.3	0.0	−0.3	63.2	−0.2	0.3	0.0	63.5	−0.4	−0.3	−0.5
Stewart wheat—												
Blade	67.0	0.2	0.0	0.5	67.2	0.0	0.6	0.0	67.5	−0.5	−0.3	−0.3
Head	67.2	0.0	0.0	0.0	67.2	−0.2	0.3	0.3	67.2	−0.4	0.3	−0.2
Vantage barley—												
Blade	47.0	0.0	0.0	−0.8	47.0	0.5	−0.2	−0.5	47.8	−1.6	−2.3	−1.8
Head	46.2	−0.2	1.3	−1.0	46.0	0.5	0.0	−0.8	46.0	0.2	0.0	−3.2
Montcalm barley—												
Blade	44.5	0.0	1.5	2.0	44.0	3.0	3.0	3.8	46.2	0.6	1.0	−0.2
Head	46.5	1.0	1.5	1.3	47.5	−1.5	−1.3	−0.5	45.2	2.0	2.3	0.8
Ajax oats—												
Blade	36.2	0.6	0.8	0.6	37.0	0.0	0.0	−1.2	37.8	−0.8	−1.8	−3.0
Head	37.0	−0.2	−1.5	−0.5	36.5	0.7	−0.5	0.0	36.5	−0.7	0.0	−0.7
Exeter oats—												
Blade	36.0	−0.8	−0.5	−0.5	35.5	0.7	−0.5	−1.0	35.8	−0.3	0.0	0.4
Head	35.8	0.2	−1.3	0.7	36.2	0.0	−1.2	−0.2	35.5	−0.5	−0.5	0.5

Values for zero dosage are for control samples.

sample was lower than the minimum for the grade, this can be disregarded in these studies since for all three determinations comparisons were confined to corresponding treated and control samples within groups. Differences of 1 grade and 1 pound were arbitrarily chosen as significant for grade and bushel weight. Mean data for bushel weight and protein content are given in Tables 1 and 2. Grade data are not presented for reasons that will be apparent when they are discussed. Principal conclusions are summarized in the following paragraphs.

Grade. Except for one sample of wheat, two samples of barley, and four samples of oats, which changed by one grade, all treated samples were identical in grade to the corresponding controls. Thus there was no consistent degrading for class of grain, variety, formulation, dosage, or time of application. Grades for control samples were: Thatcher, Nos. 2 and 3 Northern; Redman, No. 2 Northern; Stewart, No. 3 C.W. Amber Durum; Vantage, No. 1 Feed; Montcalm, Nos. 2 and 3 C.W. Six-row and No. 1 Feed; Ajax, No. 3 C.W. and No. 1 Feed; and Exeter, No. 1 Feed.

Bushel weight. For the three wheat varieties, bushel weights were quite uniform, and, except for five samples, variations above or below the controls were less than 1 pound. Four of these samples were Thatcher treated with the ester and the other sample was Redman treated with the highest dosage of the amine. Barley was affected more than wheat, and reductions were more numerous for Vantage than for Montcalm. Vantage was reduced by about 2 pounds with all dosages of the ester at the blade stage and by more than 3 pounds with the highest dosage at the head stage. Montcalm was reduced by just over 1 pound in only two samples treated with the amine, but increases of 1 pound and over occurred with all dosages of the sodium salt at the head stage, of 3 to 4 pounds with all dosages of the amine at the blade stage, and of 2 pounds with 4 and 8 ounces of the ester at the head stage. The oat varieties, like the wheat varieties, were fairly uniform in bushel weight. Ajax decreased by about 1 to 3 pounds with all dosages of the ester at the blade stage and by more than 1 pound with high dosages of the other two formulations. In no sample of Exeter was the decrease greater than about 1 pound, and then only for three samples—two amine and one sodium salt treated.

Protein content. For the wheat samples, significant variations were all increases, and the highest increases resulted from the ester treatment. With the highest dosage at the head stage, increases for Thatcher and Redman were 1.0 and 0.8 per cent, and Stewart increased by significant amounts with all three dosages. Barley was, in general, more uniform in protein than the wheat, and there were only four significant variations for both varieties. For Vantage, there was an increase of 1.1 and 1.6 per cent with 8 and 16 ounces of the ester at the blade stage and of 1.7 per cent with the highest dosage at the head stage. For Montcalm there was only one significant variation; a decrease of 0.9 per cent with the highest dosage of the sodium salt at the blade stage. The oats varieties showed only two significant variations in protein and both were increases; 0.5 per cent for Ajax with the highest dosage of the ester at the head stage, and 0.8 per cent for Exeter with the lowest dosage of the amine at the head stage.

TABLE 2.—PROTEIN DATA 1948 CROP. DIFFERENCES BETWEEN 2,4-D TREATED AND CONTROL SAMPLES

Dosage, oz.	Sodium salt					Amine					Ester				
	0	4	8	16	N.D.	0	4	8	16	N.D.	0	4	8	16	N.D.
Thatcher wheat—															
Blade	15.1	−0.2	−0.1	−0.2	0.4	14.9	0.2	0.2	0.5*	0.4	15.0	0.0	0.3	0.1	0.4
Head	15.2	0.2	0.3	0.4*	0.4	15.4	−0.2	−0.3	−0.1	0.4	15.2	0.3	0.4*	1.0*	0.4
Redman wheat—															
Blade	15.2	0.2	0.3	0.4*	0.4	15.3	0.3	0.3	0.4*	0.4	15.2	0.2	0.5*	0.4*	0.4
Head	15.4	0.4*	0.4*	0.2	0.4	15.6	0.0	0.2	0.2	0.4	15.5	0.2	0.3	0.8*	0.4
Stewart wheat—															
Blade	11.8	0.0	0.1	0.1	0.4	12.0	−0.2	−0.2	−0.1	0.5	12.2	−0.1	−0.2	−0.2	0.5
Head	11.8	0.2	0.1	0.1	0.2	11.9	0.3	−0.1	0.1	0.3	11.8	0.4*	0.4*	0.9*	0.3
Vantage barley—															
Blade	11.9	0.0	0.1	−0.1	0.9	11.8	0.1	0.1	0.2	0.9	11.8	0.3	1.1*	1.6*	0.9
Head	12.4	0.1	−0.2	0.0	0.9	12.8	−0.4	−0.4	−0.2	0.9	12.5	0.2	0.0	1.7*	0.9
Montcalm barley—															
Blade	13.4	−0.3	−0.4	−0.9*	0.9	13.2	−0.6	−0.6	−0.6	0.9	13.0	0.0	0.0	0.2	0.9
Head	12.4	−0.4	−0.3	−0.7	0.9	12.1	0.2	0.2	0.3	0.9	13.0	−0.4	−0.7	0.2	0.9
Ajax oats—															
Blade	13.9	−0.1	0.0	0.0	0.7	14.0	0.2	0.2	0.6	0.7	13.9	0.4	0.3	0.1	1.0
Head	14.2	−0.1	0.0	−0.1	0.4	14.3	−0.1	0.1	0.2	0.4	14.2	−0.1	0.0	0.5*	0.4
Exeter oats—															
Blade	12.6	0.2	−0.2	0.0	1.0	12.6	−0.3	0.1	0.1	1.0	12.3	0.0	1.1	0.4	1.3
Head	12.3	0.3	0.4	0.0	0.7	12.0	0.8*	0.5	0.5	0.7	12.6	0.2	0.3	0.4	0.6

Values for zero dosage are for control samples.
N.D. = necessary difference.

1949 Samples

The 1949 study was similar in design to that made in 1948, but there were three varieties each of barley and oats instead of two, two formulations and dosages of 2,4-D instead of three, and dates of application were different. Formulations used were the amine and the ester, and dosages were 0, 4, and 8 ounces per acre of the acid equivalent. First date of application was June 2 for wheat and barley and June 4 for oats. Second date of application was June 24 for wheat and barley and June 28 for oats. On June 2 and 4, all plants were 10 to 11 inches tall. On June 24, growth stage was 2 and 4 days before heading for Thatcher and Redman, 11 days before heading for Stewart, 2 days before heading for Montcalm and O.A.C. 21, and 5 days before heading for Vantage. On June 28, growth stage was 3 and 6 days before heading for Vanguard and Exeter, and just beginning to head for Ajax. Dates of application are again referred to as the blade and head stages.

TABLE 3.—BUSHEL WEIGHT AND PROTEIN DATA 1949 CROP. DIFFERENCES BETWEEN TREATED AND CONTROL SAMPLES

	Bushel weight, lb.						Protein, per cent					
	Amine			Ester			Amine			Ester		
Dosage, oz.	0	4	8	0	4	8	0	4	8	0	4	8
Thatcher wheat—												
Blade	64.2	0.0	−0.2	64.2	−0.2	−0.2	13.3	0.2	0.0	13.3	0.3	0.0
Head	64.0	0.2	0.0	64.0	0.5	0.5	13.7	0.1	0.1	13.7	0.3	0.3
Redman wheat—												
Blade	62.8	−0.3	−0.8	62.8	−0.6	−0.6	14.2	0.3	0.4	14.2	0.3	0.4
Head	62.5	0.5	0.7	62.5	0.7	1.0	14.4	−0.2	−0.3	14.4	0.0	−0.2
Stewart wheat—												
Blade	68.0	0.0	0.0	68.0	0.0	0.0	12.8	0.2	0.0	12.8	−0.1	0.1
Head	68.0	0.0	−0.2	68.0	−0.5	−0.8	13.2	0.1	0.3	13.2	0.2	0.3
Vantage barley—												
Blade	48.0	−0.2	−0.5	48.0	−0.2	−0.8	13.6	0.0	0.0	13.6	−0.1	0.5
Head	47.2	0.8	0.8	47.2	0.8	1.0	13.6	0.0	0.0	13.6	0.1	0.0
Montcalm barley—												
Blade	*	*	*	*	*	*	12.4	0.0	0.0	12.4	0.0	0.0
Head	47.2	1.6	1.0	47.2	1.8	1.0	12.5	0.2	0.1	12.5	0.3	0.4
O.A.C. 21 barley—												
Blade	44.2	0.3	0.0	44.2	−0.2	−1.2	13.8	−0.6	−0.5	13.8	−0.5	−0.4
Head	43.8	0.4	1.0	43.8	1.2	0.7	13.4	0.1	0.1	13.4	0.1	0.5
Ajax oats—												
Blade	38.0	0.8	0.8	38.0	−0.2	−1.8	15.1	−0.1	0.7	15.1	1.2	1.2
Head	38.0	0.8	−0.5	38.0	0.0	0.2	15.3	−0.3	0.4	15.3	0.5	0.3
Exeter oats—												
Blade	37.0	0.0	0.5	37.0	−0.8	−2.5	13.7	0.4	0.7	13.7	1.1	1.2
Head	37.2	−0.2	−0.2	37.2	1.0	1.0	13.9	0.1	0.1	13.9	0.2	0.9
Vanguard oats—												
Blade	36.2	0.3	−2.0	36.2	−3.0	−3.7	15.4	0.4	1.2	15.4	1.2	1.3
Head	36.2	−0.2	0.0	36.2	−0.2	0.6	15.3	−0.3	0.3	15.3	−0.3	−0.1

Values for zero dosage are for control samples.
*Samples too small for bushel weight determinations.

The samples submitted to the Laboratory were composites representing each quadruplicate rod row. Grade and bushel weight were determined as in the former study, and the same significant differences were used. Protein determinations were confined to duplicates of each composite sample; hence it was not possible to calculate significant differences for varieties and treatments. Since these values were obtained for the 1948 samples, they serve as a guide in this discussion. Data for bushel weight and protein content are given in Table 3. Principal conclusions are summarized in the following paragraphs.

Grade. Except for two samples of Vanguard oats treated with both dosages of the ester which changed in grade from 1 Feed to 2 Feed, and a sample of Exeter oats treated with the higher dosage of the ester which also changed in grade from 1 Feed to 2 Feed, all treated samples were identical in grade to the corresponding controls, thus confirming the results of the 1948 study. Grades for control samples were: Thatcher, No. 2 Northern; Redman, No. 2 Northern; Stewart, No. 1 C.W. Amber Durum; Vantage, No. 1 Feed; Montcalm, No. 2 C.W. Six-row; O.A.C. 21, No. 3 C.W. Six-row; Ajax, No. 3 C.W.; Exeter, No. 1 Feed; and Vanguard, No. 1 Feed.

Bushel weight. With one exception, all samples of wheat varied by less than 1 pound in bushel weight from the controls. The same is true for Vantage barley and Ajax oats. Montcalm increased by 1 to almost 2 pounds with both dosages of the amine and the ester, and O.A.C. 21 increased by 1 pound with the higher dosage of the amine, and there was an increase and a decrease of over 1 pound with the ester. Exeter was not affected by the amine, but was reduced by over 2 pounds with the higher dosage of the ester at the blade stage, and increased by 1 pound with both dosages at the head stage. Vanguard was affected more than Exeter. It was reduced by 2 pounds with the higher dosage of the amine and by 3 to almost 4 pounds with both dosages of the ester at the blade stage.

Protein content. When compared with controls, variations in protein content of the treated samples were mostly increases, but in only comparatively few samples were these increases considered significant. For the wheat varieties, no difference exceeded 0.4 per cent, and for the barley varieties no difference exceeded 0.6 per cent. For the oats varieties, there were several increases in protein of over 1 per cent and these values are probably significant. Such increases were obtained for both dosages of the ester at the blade stage. Slightly smaller increases were also found for oats with the higher dosage of the amine at the blade stage.

Discussion

The results of these two studies show that the varieties of wheat, barley, and oats studied were not consistently lowered in grade by either the sodium salt, the amine, or the ester formulations of 2,4-D, even when dosages higher than those recommended were used. The bushel weight data are inconclusive since sometimes the treated samples showed increases and sometimes decreases. Wheat appeared to be affected least and reductions were more numerous for the ester-treated samples.

The protein data for the treated samples show that variations from the controls were often increases, though these increases were generally not significant. In the 1948 study, in which sufficient data were available to calculate significant differences, increases were greater for wheat than for the other grains, and most pronounced for the ester formulation.

It is difficult to generalize about the increases in protein content which occurred as an apparent result of 2,4-D treatments, since when this is obtained without a decrease in bushel weight it may be an advantage, and when obtained at the expense of bushel weight it may be a disadvantage. For bread wheat and oats, high protein is obviously desirable, and, within limits, for macaroni wheats also. But for malting barley an increase in protein content usually results in a decrease in yield of malt extract, and hence is a disadvantage.

PART II—GENERAL QUALITIES OF BREAD WHEAT AND MALTING BARLEY

This part of the paper deals with more extensive quality tests made on bread wheat treated with 2,4-D in 1949, and on barley treated in 1948 and 1949. The samples were from experimental plots used by the Department of Plant Science, University of Manitoba, in investigations of the effect of 2,4-D on yield per acre, etc. The samples represented plots in which yields varied quite widely, and were studied by the Laboratory to obtain information on the effect on the milling, baking, and dough qualities of wheat and the malting properties of barley when 2,4-D had been applied at different stages of plant growth. The tests made were those normally used for varieties submitted by plant breeders.

Bread Wheat

This set of bread wheats comprised 10 composite samples representing replicated plots of Redman which had been sprayed with dosages corresponding to 12 ounces per acre of the pure acid of the ester formulation of 2,4-D on the dates shown in Table 4. Heights of plants at time of treatment are also given in the table. Grades were assigned by the Winnipeg Inspection Office.

After determinations of bushel weight and protein content, 1500-g. samples were milled in the laboratory Allis-Chalmers mill. Larger samples are usually milled; hence too much importance should not be attached to flour yield. The flours were analysed for protein and yellow pigment contents, and were baked by the malt-phosphate-bromate procedure. Mixing and stretching curves—which indicate such physical properties of dough as ability to withstand prolonged mixing and extensibility and resistance to stretching—were made in the farinograph and the extensograph. Normal flour-water doughs were used in both machines at predetermined levels of absorption, and in the extensograph, a second series of curves was made to show the effect of the addition of 0.003 per cent potassium bromate.

Results of the milling and chemical tests, the more important baking data, and customary measurements taken from farinograms and extensograms are given in Table 4. Data for the treated samples are given as differences from the untreated control sample.

TABLE 4.—QUALITY DATA FOR WHEAT 1949 CROP. DIFFERENCES BETWEEN 2,4-D TREATED SAMPLES AND CONTROL

Date of application	Control	May 10	May 13	May 16	May 19	May 23	May 30	June 13	June 16	June 20
Growth stage, inch	—	1	2-3	4	5	5.5	6	14	17	18
Grade	2Nor.	2Nor.	2Nor.	3Nor.	3Nor.	3Nor.	2Nor.	2Nor.	2Nor.	2Nor.
Bushel weight, lb.	62.5	0.0	−0.5	−0.5	−1.3	−0.5	0.0	1.0	1.5	2.0
Flour yield, %	70.2	0.2	0.3	0.2	−0.9	−3.7	0.5	0.1	0.3	1.0
Wheat protein, %	15.8	−0.2	0.2	−0.2	0.5	0.4	−0.1	0.1	0.4	−0.1
Flour protein, %	14.9	0.0	0.1	0.0	0.8	0.7	0.1	0.3	0.7	0.1
Flour pigment, p.p.m.	2.18	0.04	−0.06	−0.04	−0.06	−0.11	−0.04	−0.05	−0.12	−0.09
Baking data—										
Absorption, %	61.0	0.4	1.2	2.8	1.7	1.8	0.9	1.1	1.6	1.7
Loaf volume, cc.	820	−15	20	10	60	−25	−40	10	10	0
Crumb texture	6.5	0.0	0.0	0.0	0.0	0.0	0.0	0.5	0.5	0.0
Crumb colour	7.0	0.0	0.5	0.0	0.5	−0.5	−0.5	0.5	1.0	0.5
Farinogram—										
Development, time, min.	6.5	0.25	1.75	0.25	0.25	−1.5	−1.0	0.0	−0.5	−0.5
Extensogram—										
Length, cm.:										
No bromate	23.9	−0.3	0.4	1.0	−1.6	−2.4	−2.9	−0.4	−0.4	−1.0
0.003 % bromate	20.8	−0.2	−0.9	0.6	−0.3	1.4	−1.1	−0.2	−0.2	−0.2
Height, cm.:										
No bromate	6.9	−0.7	−0.9	−1.2	−1.5	−2.3	−0.9	−0.2	−1.1	−1.4
0.003 % bromate	9.4	−0.5	−1.0	−1.2	−1.8	−2.8	−1.1	0.2	−1.3	−1.2

The data are not particularly clear-cut, since a lowering in one quality of some of the treated samples is sometimes accompanied by improvement in another quality. Nevertheless, the data do indicate that the wheats treated when plant height was about 4 to 6 inches differ from the control in some qualities more than the wheats treated when plant height was 1 to 3 inches or 14 inches and taller. (At 4 inches, the plant was in the two-leaf stage, tillering occurred at 6 inches, and 14 inches was about two weeks before heading). For example, the wheats treated when plant height was 4, 5, and 5.5 inches were a grade lower, 0.5 to 1.3 pounds lower in bushel weight, quite variable in protein (0.2 per cent lower to 0.5 per cent higher), and 1.7 to 2.8 per cent higher in absorption. The largest and the two smallest loaves were for the samples treated when plant height was 5, 5.5, and 6 inches, and the two smallest loaves were poorest in crumb colour. All doughs were, however, quite satisfactory in handling quality. The mixing and stretching curves lend support to the baking data, since the two samples which gave the smallest loaves also gave the least desirable farinograms and doughs with the least extensibility.

Samples treated when plants were 14 inches and taller (one to two weeks before heading), were not much different from the control in protein content, loaf volume, and type of farinogram and extensogram. In general, the same observations apply to the wheat treated at the beginning of the season when plants were quite small.

Discussion

While the data for this set of Redman samples do not provide very definite information on the quality of wheat treated at different stages of plant growth with the ester formulation of 2,4-D, there is an indication that quality is affected least when spraying was done when plants were quite small (about 2 inches) or when growth was well advanced (14 inches or taller). When sprayed at an intermediate growth stage (just before or at tillering), there were greater variations from the control in some qualities. None of these differences in quality is particularly serious, but they seem to indicate that there is a fairly critical period in the time of 2,4-D application when grade, bushel weight, and baking quality may be affected.

Data reported by Olson *et al.* (8) show that only two of the lots of wheat tested by the Laboratory suffered significant reductions in yield per acre as a result of the 2,4-D treatment; one lot when growth was 5 inches (about a week before tillering), and the other lot when growth was 17 inches (about two weeks before heading). The first of these samples, which had the lowest yield per acre of the series (31.9 bushels as compared with 40.6 for the control), had the highest protein content and gave the largest loaf, though it had the lowest bushel weight and suffered a loss in grade. On the other hand, the sample which gave the smallest loaf (6-inch, about the tillering stage) was closer to the control in yield per acre (39.9 bushels) than all but one of the samples tested. It would thus seem that improved baking strength was obtained at the expense of yield per acre, and vice versa.

Malting Barley

Although three separate studies were made on samples of malting barley, two of them were more or less preliminary, though extensive, and the results are discussed without presenting data. The third and more important study closely paralleled that made on Redman wheat, and the data are presented and discussed in more detail.

Preliminary Studies

Additional quality tests were made on the barley samples from the 1948 crop mentioned in *Part I* and listed in Table 2. These additional tests were: Percentage heavy grade barley; germination of heavy and medium grade fractions; and malting tests on the heavy grade portions. Statistical analyses showed that there were significant interactions between varieties, dates of treatment, formulations, and dosages, so that significant effects of 2,4-D treatment occurred only within certain sets of treatments. For example, yield of heavy grade barley of Vantage was not affected by any 2,4-D treatment, but all formulations increased yield of heavy grade barley of Montcalm. The increase varied with dosage, formulation and date of application. When applied when the plants were 6 inches high (blade stage) all dosages of the amine and ester increased heavy grade of Montcalm by 12 per cent, but only the one pound dosage of the salt was effective to this degree. All formulations increased heavy grade of Montcalm when applied just before heading, and the increase was progressive with increase in dosage to a maximum of 10 per cent.

The germination of the heavy grade portion of both varieties was not affected by 2,4-D treatment, but the medium size kernels were reduced in germination by the amine and ester. The damage was greater for Vantage than for Montcalm, and increased with increase in dosage.

The effects of 2,4-D on the malt properties of the barley were also variable. The malt extract of Montcalm was not affected by most treatments, but the heavy application of the amine when plants were at the blade stage increased extract by 1.5 per cent and all dosages of the salt applied before heading increased extract by 1 per cent. On the other hand, with Vantage, the heavy dosage of amine at the first date decreased extract content by 2 per cent; all dosages of the ester applied at the first date reduced extract by 1 per cent; and the one-pound dosage of the ester at the second date reduced extract by 2 per cent. Other properties were not significantly affected by any treatment.

The results of 2,4-D treatment are irregular, and therefore unpredictable. The most obvious feature of the data was that Vantage was adversely affected by some treatments, while significant effects on Montcalm tended to improve malting quality. The adverse effects on the malting properties of Vantage, which is not a malting variety, were accompanied by obvious defects in the appearance of the kernels, which resulted in lowering of grade. From the practical aspect, this is important and indicated that 2,4-D damage could be ascertained during grading of samples. Nevertheless, further experiments on malting varieties were planned for the succeeding year.

A same study was made in 1949 to obtain information on the comparative effects of the amine and ester formulations of 2,4-D on barley quality when applied on two different dates to plots of O.A.C. 21 and Montcalm. The samples were controls and corresponding samples treated with 4 and 8 ounces of the acid equivalent per acre of each formulation applied when plant growth was 6 inches and when plants were at the near-heading stage. The samples were malted and analysed by the standard methods of the Laboratory, and included determinations of percentage heavy grade kernels, kernel weight, barley nitrogen content, yield of malt extract, saccharifying activity, and wort nitrogen of malt.

The 2,4-D treatments applied when the plants were 6 inches tall had no marked effect on the barley properties of either variety, although the 4-ounce ester treatment tended to reduce the percentage of heavy grade barley suitable for malting. All treatments, however, did increase the malt extract of Montcalm but not that of O.A.C. 21. Effects on saccharifying activity and wort nitrogen were not significant.

The results of the 2,4-D treatments when the plants were at the near-heading stage differed from those when growth was 6 inches. All treatments at this later stage of development increased the percentage of heavy barley, and the ester formulation increased kernel weight also. These increases were found for both varieties. All treatments tended to increase malt extract yield of Montcalm, but there were no significant effects of treatments on any other malt property. These two studies, made in different years, agree in essential features. A differential response of varieties to 2,4-D treatment is evident, but the quality of O.A.C. 21 and Montcalm, the principal malting varieties in Canada, was not adversely affected by the treatments used.

Study on Montcalm

This set of samples consisted of 12 composites representing replicated plots of Montcalm barley grown in 1949 that had been sprayed with dosages corresponding to 12 ounces of the pure acid of the ester formulation of 2,4-D on the dates shown in Table 5. The samples were selected from a larger series so that the critical and non-critical dates of application, as judged by variations in nitrogen content, were represented. Heights of plants at time of treatment are also given in the table. Grades were assigned by the Winnipeg Inspection Office. The samples were analysed and malted by the same methods used in the preliminary studies. As in the Redman wheat study, data for the treated samples are given as differences from the control sample.

The prominent feature of the data is that the barley treated on June 20, when plant growth was 6 days before heading, differed from the control in certain important qualities more than any other sample. It was highest in nitrogen content, lowest in malt extract, and one of the highest in saccharifying activity. In addition to these important malt properties, the sample was lowest in grade and bushel weight, as well as being definitely lowest in yield per acre (15.3 bushels). The other samples are judged to be at least equal to the control in general malting quality and many are

TABLE 5.—QUALITY DATA FOR BARLEY 1949 CROP. DIFFERENCES BETWEEN 2,4-D TREATED SAMPLES AND CONTROL

Date of application	Control	May 10	May 13	May 16	May 19	June 2	June 4	June 7	June 16	June 20	June 23	June 27
Growth stage, inch	—	1	2	4	4.5	8	10	12	17	18	18-20	18-20
Barley properties—												
Grade*	2 C.W. 6	2 C.W. 6	2 C.W. 6	3 C.W. 6	3 C.W. 6	2 C.W. 6	3 C.W. 6	3 C.W. 6	2 C.W. 6	1 Feed	3 C.W. 6	2 C.W. 6
Bushel weight, lb.	47.8	0.2	0.4	2.2	2.0	0.4	0.2	−0.3	1.4	−2.3	2.2	1.4
Heavy grade, %	74.2	6.0	8.2	18.7	18.5	4.2	4.0	−4.9	14.4	17.6	22.5	12.0
100 kernel weight, gm.	32.6	−0.2	1.5	2.6	3.2	−0.4	−1.0	−1.4	1.2	3.1	4.1	2.3
Nitrogen, %	2.31	−0.03	0.05	0.12	0.14	0.00	−0.07	−0.04	0.07	0.30	0.20	0.13
Malt properties—												
Extract, %	76.1	−0.4	−0.6	0.4	0.2	0.1	0.3	0.0	−0.4	−2.0	0.5	−0.2
Wort N., %	1.24	−0.05	0.04	0.04	0.05	0.03	−0.04	−0.02	0.00	0.14	0.10	0.04
Sacch. Act. ° L.	183	−9	32	25	17	22	1	13	12	31	30	9

*6 = Six-row.

better in some properties. There are, however, some effects of the 2,4-D treatment on barley and malt properties of the series as a whole that are worthy of mention.

The increases in nitrogen content of barley treated between May 13 and 19, when plant growth was 4.5 inches or less (tillering occurred at 7 inches), and between June 16 and 27, when plant growth was 17 inches and taller (heading occurred at 18-20 inches) are accompanied by increases in percentage heavy grade kernels and kernel weight. This is contrary to the usual association between low kernel weight with high nitrogen content. The increase in kernel weight with nitrogen content is reflected in the maintenance of malt extract yield, which, except for the sample treated on June 20, varied by less than 1 per cent from that of the control. Except for the one sample treated on May 10 when the plant was barely above ground, values for saccharifying activity are higher for all treated samples. Higher nitrogen content accounts for part of these increases, but they are higher than would be expected. This suggests that the ester formulation of 2,4-D had some effect on the enzyme system of the grain. Data for wort nitrogen lend support to this assumption, since, in general, the values are lower than would be expected when comparison is made with those for barley nitrogen.

Discussion

The results of these malting tests for this set of Montcalm barley show that the quality of the mature barley is affected by treatment with the ester formulation of 2,4-D at some stages of plant growth, and it is apparent that metabolic disturbances occurred in the growing plants. Some of the customary relationships between certain barley and malt properties are changed and others are not; hence the effects are not yet predictable. For example, increases in barley nitrogen content, which in terms of protein are well over 1 per cent for some samples, were not always accompanied by corresponding decreases in malt yield, which is usual; and, except for a single sample, malt extract yield was fairly uniform.

When the malting data are considered in the same way as the data in the Redman wheat series, they show greater variations from the control when plants were treated between just after emergence and well ahead of tillering (2 to 4.5 inches) or near heading time (18 to 20 inches) than at an intermediate growth stage. In these two periods, saccharifying activity was definitely higher, nitrogen content was higher, and malt extract was lowest and next lowest for one sample in each period. Sometimes grade was reduced also.

Data reported by Olson *et al.* (8) show that reductions in yield of barley per acre were significant for all but three of the samples tested, and two of these samples (8 and 10 inches) are in the intermediate group referred to above. The yields for the other two samples in this group, though significantly reduced, were much higher than those for the samples treated when plant height was 18 inches and more. It would seem, therefore, that application of 2,4-D at an intermediate growth stage (after tillering and well before heading) is the most satisfactory time from the standpoint of both yield per acre and quality.

General Discussion

Though the growing time from plant emergence to about a week before heading was the same for the Redman wheat and Montcalm barley, strict comparisons to determine the best time for the 2,4-D application cannot be made, because dates of application were not always the same for both sets of samples. Nevertheless, there is evidence of a critical time for each grain and these times are not the same. For Redman wheat, yield per acre was lowest when plants were 5 inches tall when treated, but baking quality was improved; treatment at 6 inches did not affect yield but reduced baking quality; and treatment at 18 inches (7 days before heading) did not affect either yield or baking quality. For Montcalm barley, yield per acre and malting properties were both satisfactory when plants were 8 inches tall (just after tillering) when treated, but treatment at 18 inches (6 days before heading) reduced yield per acre from 80.7 to 15.3 bushels and malting properties were not satisfactory. Thus the "best" time for 2,4-D treatment of wheat is not necessarily the "best" time for barley, and vice versa. Treatments on barley that seriously reduce yield also impair quality; but for wheat, treatment at certain stages of plant growth affect yield and quality in opposite directions. Whether these observations would hold true for other varieties of wheat and barley or in similar studies made in other years, must, of course, await further investigation.

SUMMARY

Quality data for typical varieties of Western Canadian wheat, barley, and oats grown at one location and treated with different formulations and dosages of 2,4-D at various stages of plant growth are presented in two parts. *Part I* deals with the effect of the sodium salt, amine, and ester formulations on grade, bushel weight, and protein content of different varieties grown in 1948 and 1949. *Part II* deals principally with the effect of the ester formulation on the baking and dough properties of Redman wheat and on the malting properties of Montcalm barley.

Part I data show that the varieties of wheat, barley, and oats, were not consistently lowered in grade by either of the 2,4-D formulations. Bushel weight data were inconclusive since sometimes treated samples showed increases and sometimes decreases. Variations from controls in the protein content of treated samples were often increases. These increases, though not always significant, appear to be greater for wheat than for the other grains, and more pronounced for the ester formulation.

Part II data indicate that there is a time during the growing period when application of the ester formulation of 2,4-D may cause changes in the quality of wheat and barley and that these times are not the same for both grains. Treatments on barley that seriously reduce yield also impair quality, but for wheat, treatments at certain stages of plant growth affect yield and quality in opposite directions. Whether these observations would apply to varieties other than Redman wheat and Montcalm barley, or to similar studies made in other years, must await further investigation.

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THE REVIVAL OF THE NARCISSUS BULB EELWORM, *DITYLENCHUS DIPSACI* (KUHN) FILIPJEV, FROM SUBLETHAL HOT WATER TREATMENTS[1]

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INTRODUCTION

The susceptibility of the narcissus bulb eelworm to destruction by heat is one of the principles on which the hot water treatment for the control of eelworms in bulbs is based. However, conflicting statements have appeared in respect to the time required to kill this eelworm at 110° F. In 1933, Staniland (6) claimed that eelworms from fresh narcissus leaves could be killed in 17 minutes when they were immersed in a water bath at 110° F., but, in the same year, Hastings (2) pointed out that eelworms occurring as eelworm wool in narcissus bulbs could not be killed at that temperature in less than 110 minutes. In 1934, Sherman (5) reported that eelworms in fresh narcissus leaves were less resistant than those in dry leaves, and that the populations in the former were composed of adults and young larvae, whereas those in the latter consisted entirely of pre-adults. In the same year, Hastings and Newton (4) found that the pre-adult is the most resistant stage in the life of this eelworm and that eelworm wool is composed mainly of pre-adults. Thus it would seem that the stage in the life cycle of the narcissus bulb eelworm as well as its condition, whether active or dormant, are factors which contribute to the wide differences in the reports upon the lethal temperature-time factor.

The statements in the literature in regard to the lethal temperature-time factor are also somewhat misleading, owing to a lack of agreement in respect to the symptomatology of death. Most investigators accept the recovery of motility by eelworms within a few days after their subjection to heat treatment as evidence of life, and lack of motility as evidence of death. It may be pointed out, however, that, although the movement of normal eelworms is serpentine, those injured by heat may exhibit only a slight bending or twitching movement for some time before death. Those that exhibit the normal serpentine movement and those that only twitch are obviously not in the same class. Furthermore, a time limit for the recovery of motility after treatment has not been established or standardized. The need for a standard technique in eelworm survival studies is all the more apparent when it is realized that many factors are known to induce quiéscence or to delay recovery of motility (1, 3), factors that may lead to the erroneous classification of living eelworms as dead ones.

The present investigation was an attempt to establish a time limit for the revival of the narcissus bulb eelworm after subjection to the hot water treatment. In it, the recovery by the eelworm of a normal serpentine movement only was regarded as evidence of survival.

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EXPERIMENTAL

Influence of age of eelworm wool on eelworm survival

Eelworm wool, stored 25, 40, and 376 days after collection from narcissus bulbs, was placed in tap water in shallow dishes over night to induce the worms to become motile. Portions of each of these eelworm suspensions, 2 ml. in volume, were placed in test-tubes. Each series consisted of three test-tubes divided into three sets. One set of each series was placed in the water bath at 110° F. for one hour; another set for two hours, and the remaining one for three hours. The experiment was repeated. Test tubes containing eelworms of the 376-day-old sample were kept in the bath for four and for five hours. As each test-tube was removed from the bath, the contents were decanted into a Syracuse dish, where a large air surface and a shallow substrate favoured revival. Revival data were taken on the 2nd, 6th and 10th day after treatment. The results are summarized in Table 1.

TABLE 1.—THE PERCENTAGE OF EELWORMS SHOWING RECOVERY OF MOTILITY AFTER HOT WATER TREATMENT AT 110° F.

Duration of treatment	Percentage of motile eelworms								
	25-day-old			40-day-old			376-day-old		
	2nd day	6th day	10th day	2nd day	6th day	10th day	2nd day	6th day	10th day
One hour	82	97	89	76	88	82	2	46	46
Two hours	21	94	90	16	66	65	0	39	39
Three hours	16	87	87	12	66	70	0	2	2
Four hours	—	—	—	—	—	—	0	0	0
Five hours	—	—	—	—	—	—	0	0	0

It is evident from Table 1 that the resistance of the worms to this heat treatment becomes progressively lower as the eelworm wool ages. In the 376-day-old sample, the percentage of eelworms that recovered was lower, and their recovery was much slower, than in the 40-day-old and the 25-day-old sample. It is also evident that the recovery of motility by the eelworm is progressively slower as the length of the heat treatment increases. For example, on the second day after treatment, eelworms of the 25-day-old sample subjected to a 3-hour treatment showed a recovery of 16 per cent., whereas those subjected to a 1-hour treatment showed a recovery of 82 per cent. However, more recoveries continued to take place daily, and reached a maximum either before or on the 6th day, for observations made on the 10th day did not differ greatly from those made on the 6th day. It is concluded that all eelworms capable of recovery will have recovered within six days after the treatment.

Effect on eelworm recovery of two hot water treatments

The effect on the bulb eelworm of two hot water treatments does not appear to have been investigated. A comparison was, therefore, made of the recovery of motility by eelworms given a single immersion at 110° F. and those subjected to a second immersion one week after the first. The results of the experiment are summarized in Table 2.

TABLE 2.—THE PERCENTAGE OF EELWORMS SHOWING RECOVERY OF MOTILITY FROM ONE AND FROM TWO TREATMENTS

Duration of treatment	Number of treatments	Percentage motile					
		2nd day	3rd day	4th day	5th day	6th day	10th day
One hour	One	66	71	78	77	76	—
"	Two	74	78	78	73	68	—
Two hours	One	12	24	76	68	76	—
"	Two	26	36	75	73	73	—
Three hours	One	0	2	13	25	57	46
"	Two	3	14	28	56	56	48
Four hours	One	0	0	3	4	24	23
Five hours	One	0	0	2	2	8	8

As shown in Table 2, the percentage of eelworms recovering motility from one treatment differed only slightly from the percentage recovering from two treatments. For instance, recovery from a three-hour treatment was 57 per cent and 56 per cent for one treatment and two treatments respectively. The first treatment resulted in the death of 43 per cent of the population, but a second treatment only increased the death toll to 44 per cent. Therefore, nearly all of the eelworms that survived the first treatment were resistant to the second treatment. However, eelworms that received two successive treatments made a more rapid recovery after the second treatment than after the first treatment, and the more severe the treatment the greater was the difference in the speed of recovery. The recovery by the eelworms that were given two successive immersions at 110° F. reached its peak on the third day after the one-hour treatment, on the fourth day after the two-hour treatment, and on the fifth day after the three-hour treatment. Maximum recovery by the eelworms that received one treatment was one day later in each case. If the rate of recovery is regarded as an indication of eelworm resistance, then it must be concluded that the nematodes that recover from a single hot water treatment are more heat-resistant than the original population.

These results may have a bearing on a common practice in the hot water treatment of bulbs. When narcissus bulbs are immersed in a bath heated to 110° F., the temperature may be lowered by 8 or 10 degrees through the absorption of heat by the bulbs. The time that the bulbs are in the bath when the temperature is lower than 110° F. is not counted as a part of the hot water treatment, and it may be one or two hours before the temperature of the bath is raised to 110° F. again. One or two hours at the sublethal temperature may have an effect similar to that of a second treatment.

Resistance of dry dormant eelworms existing as eelworm wool to heat

Since it is an established fact that dry dormant eelworms existing as eelworm wool (2, 3, 4) are highly resistant to the temperature used in the hot water treatmeut, their recovery from sublethal treatments should be

rapid. Portions of eelworm wool were placed in test-tubes and the test-tubes were immersed in a water bath. After being subjected to the heat treatment, the pieces of wool were placed in Syracuse dishes and 2 ml. of water were added to each dish to allow the nematodes that survived to become motile. The evidence obtained showed that even a five-hour treatment at 110° F. of dry eelworms in the condition of eelworm wool, did not approach being a lethal treatment. The recovery of motility by these eelworms was, therefore, always rapid and reached a maximum on the second day after treatment.

TABLE 3.—THE PERCENTAGE OF EELWORMS SHOWING NORMAL MOTILITY AFTER HEAT TREATMENT AT 110° F.

Duration of treatment	Percentage motile		
	2nd day	3rd day	6th day
One hour	100	100	98
Two "	100	98	98
Three "	97	98	86
Four "	95	96	95
Five "	90	90	88

SUMMARY

Eelworms comprising the eelworm wool of narcissus bulbs become, with age, less resistant to hot water treatment. Both the rate of recovery and the percentage of eelworms that recovered were lower in a 376-day-old eelworm sample than in a 40-day-old and a 25-day-old sample. There appears to be a direct relationship between the severity of injury and the time required to revive heat-injured eelworms. Eelworms given a one-hour immersion made maximum recovery in approximately four days; those given a two-hour immersion, in five days; and those given a three-hour immersion, in six days. Recovery by eelworms that received two successive immersions, one week apart, was more rapid after the second immersion than after the first. Eelworms in the dry eelworm wool condition were highly resistant to heat treatment and made maximum recovery on the second day after treatment, even when the treatment was of five-hour duration.

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ALUMINIUM VERSUS TINNED-STEEL MILK CANS— A COMPARATIVE STUDY

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It is well known that milk quality is endangered during collection and processing and that the extent of quality change depends partially upon the utensils and processing equipment with which the milk comes in contact. Because milk and cream remain in contact with the can for a longer period of time than with any other piece of equipment, the milk can is a very important link in the quality chain. Any weakness in this link will be manifested in the finished product.

The conventional tinned-steel types of cans are recognized as not being entirely satisfactory in so far as they rust very quickly and hence are in need of frequent retinning. Open seams are also troublesome, and many investigators have shown that open seams and rust spots make the cleaning of milk cans more difficult.[4]

Aluminium cans, on the other hand, because of the nature of the metal, do not rust. Methods of manufacture of aluminium cans, such as spinning or drawing, give the can the advantage of being seamless. Because of these facts a comparative study of aluminium and tinned-steel cans seemed warranted.

The purpose of this investigation was to determine:

(1) The effect of milk and cream on aluminium and aluminium alloys.

(2) The effect upon the quality of milk and cream when stored in aluminium and tinned-steel cans.

(3) The corrosion rates of aluminium and tinned-steel resulting from cleaning and sterilizing solutions.

(4) The numbers of bacteria remaining in aluminium and tinned-steel cans after cleaning.

(5) The effect of commercial service on aluminium and tinned-steel cans.

REVIEW OF LITERATURE

The literature pertaining to this investigation is so voluminous that reference will be made only to those reports which are considered pertinent to this study.

In 1932 Gebhardt and Sommer (6) investigated the action of milk on metals. With regard to the effect of milk on aluminium and tinned-steel they found that the metals were very resistent to corrosion by fresh milk in the cold, and at pasteurization and boiling temperatures, and that free access to air did not promote their corrosion. However, in the case of

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[4] Seamless tinned-steel milk cans were introduced on the market when the work reported in this paper was nearing completion.

tinned-steel, which had the base material exposed, they found that the exposed portions might be rapidly attacked. Investigations conducted concerning the effect of the acidity of milk showed that it has no effect at 20° C., but at 70° C. and at 97° C. corrosion increases with an increase in acidity. Numerous other investigators before and since that time have simply confirmed these findings.

Hunziker *et al.* (7) in 1929 conducted a study on the effects of different metals on the flavour of milk and milk products. They found that aluminium and tin do not produce off-flavours unless they are taken up in large amounts, whereas exposed iron (as in rusty cans) very readily impairs the flavour of milk and milk products.

When most common metals are exposed to the atmosphere or to aqueous solutions, they become covered with a thin invisible oxide film. In the case of aluminium, the film is highly protective against further oxidation so that it never attains an appreciable thickness. In the case of some other metals, particularly iron, oxidation will continue indefinitely forming heavy scale-like layers (1). Any solution attacking this oxide film will naturally be corrosive to the underlying metal.

In the past, can washing solutions have been alkaline in nature. Most alkaline washing solutions are corrosive to most metals (the notable exception being NaOH and steel). Corrosion by alkaline solutions may often be inhibited by the addition of sodium metasilicate. The notable exception again is NaOH which cannot be inhibited by sodium metasilicate (3). More recently acid can washing solutions have been placed on the market. Parker (9, 10) found several of them to be non-corrosive to metals. Botham and Dummet (3) in 1949 investigated the corrosion of metals by hypochlorites and found that the addition of 0.25 per cent by volume of sodium metasilicate to hypochlorite diluted to 150 p.p.m. available chlorine reduced attack on all metals to the vanishing-point except aluminium which was slightly attacked at the edges after 24 hours.

In order to avoid bacterial contamination of milk, Russel (12) in 1897 advised that cans containing rust spots, patches or open seams should not be used for the transportation and storage of milk. Since that time numerous investigators have elaborated upon that recommendation.

The useful length of service of a tinned-steel milk can is recognized as being very short. Pannel (8) points out that the duration of service given by a tinned-steel can before retinning is at most five years, whereas aluminium cans last at least ten years.

EXPERIMENTAL METHODS AND EQUIPMENT

Corrosion Tests with Milk Products

Super purity aluminium and aluminium alloys 1S $\frac{3}{4}$ H, 2S $\frac{3}{4}$ H, 57S $\frac{1}{2}$ H, 65ST and Anticordal B were investigated. Circular discs of the above-mentioned alloys, having a surface area of 3.49 square decimeters, were fully immersed in sweet whole milk for 24 hours and acid cream for 72 hours at room temperatures (20°–25° C.). During the tests the discs were held in plastic corrosion spools which insulated them from each other and from the walls of the three-gallon container. The milks used had a fat percentage of 3.5–4.0 per cent and the creams 30–40 per cent. The acidity of the milks at the beginning of the tests was 0.17 per cent and increased to

PLATE I—VARIOUS TYPES OF CANS

Left to right Danish type aluminium, Canadian type aluminium, tinned-steel (seams), tinned-steel (seamless).

0.21–0.23 per cent during the tests. In the case of the creams the original acidity was 0.12 to 0.15 per cent which increased to 0.70–0.80 per cent during the tests. All tests were repeated five times using the same discs. The test discs, before and after each test, were brushed under running water, rinsed with distilled water, swabbed with ethyl alcohol, swabbed again with ethyl ether, dried 24 hours in a desiccator and weighed.

Corrosion Tests with Cleaning and Sterilizing Solutions

Strips of aluminium (alloy 2S $\frac{3}{4}$ H) and hot dipped tinned-steel (half of which had the base material exposed on the edges in order to simulate rusty can conditions) were used in these tests. Each strip had a surface area of 0.405 square decimeters. The strips were treated, before and after each test, in a similar manner as in the corrosion tests with milk. Alkaline solutions were prepared at a strength of 0.4 per cent, acid solutions at pH 4, and chlorine solutions at 100 p.p.m. available chlorine. The method of conducting the corrosion tests was to place 200 ml. of the cleaning or sterilizing solution in a 250 ml. beaker, raise the temperature to 60° C. $\pm$ 2° in a water bath, and fully immerse the strips in the solutions for 2 hours. Each test was completed in duplicate and the results averaged.

Storage Tests

Milks and creams, clean in flavour, were selected at the weigh tank for these tests. Three products were used in the tests, namely: sweet whole milk stored at 4° C., high acid cultured milk (0.83 per cent acid) stored at room temperature, and cream stored at room temperature. Each product was divided into three portions and one portion placed in each of three types of cans, viz., aluminium, well tinned-steel, and partially rusty tinned-steel. The cans were cleaned before the tests by revolving four times in a rotary-type can washer. Flavour determinations were made by four members of the Dairy Department staff. Iron was determined colorimetrically by the method of Ruegamer *et al.* (11) and aluminium was determined by a spectrographic method essentially that of Cholak, Hubbard and Story (4).

Types of Cans

Four types of cans were used in this study as illustrated in Plate I.

Bacteriological Methods

The rinsing of the cans, plating of the can rinses and the counting of the total number of colonies produced per can were carried out in accordance with Standard Methods for the Examination of Dairy Products (2). In order that controlled rinsing of the cans could be achieved, a can-rinsing machine illustrated in Standard Methods (2) was employed. The strong acid and caseolytic colonies produced per can were counted by a method advanced in the Difco Manual (5) except that tryptone glucose extract agar plus skimmilk instead of nutritive caseinate agar was used as the medium.

Can Temperature Measurements

Can temperatures were determined with a Lewis Pyrometer-Potentiometer Model 78PO7 with a copper-constantan thermocouple. The thermocouple was attached to the cans by means of water-resistant tape.

EXPERIMENTAL RESULTS AND DISCUSSION

Corrosion of Aluminium and its Alloys by Milk and Cream

In order that a metal be satisfactory for the fabrication of milk cans it must not be appreciably corroded by the milk products which might be transported or stored in it. Many investigators have studied the effects of milk products on tinned-steel. Hence in this study only the effects of milk products on aluminium have been undertaken.

The effects of milk and cream on aluminium and its alloys are summarized in Figures 1 and 2.

The results of the tests show that aluminium and its alloys are only negligibly attacked by the milks and creams under the conditions of the tests. An examination of Figures 1 and 2 reveals that in the initial test periods, the rates of corrosion are comparatively high while in the latter

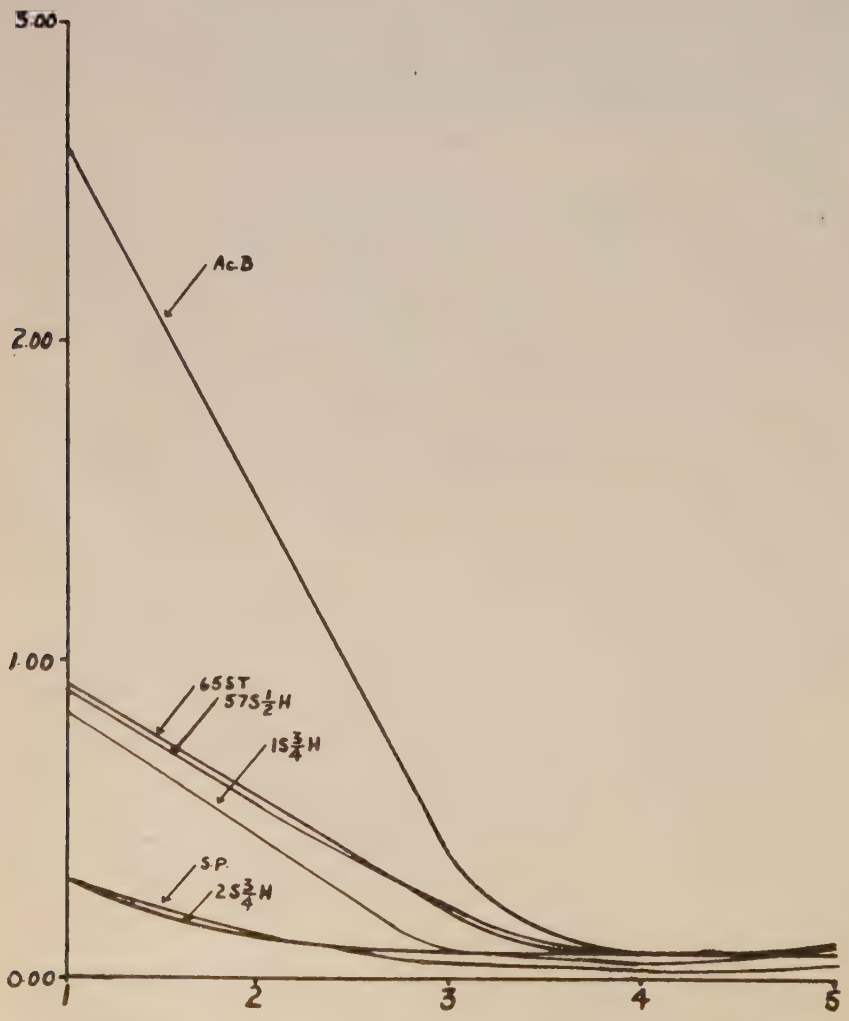

FIGURE 1. Corrosion of aluminium and aluminium alloys by sweet whole milk.

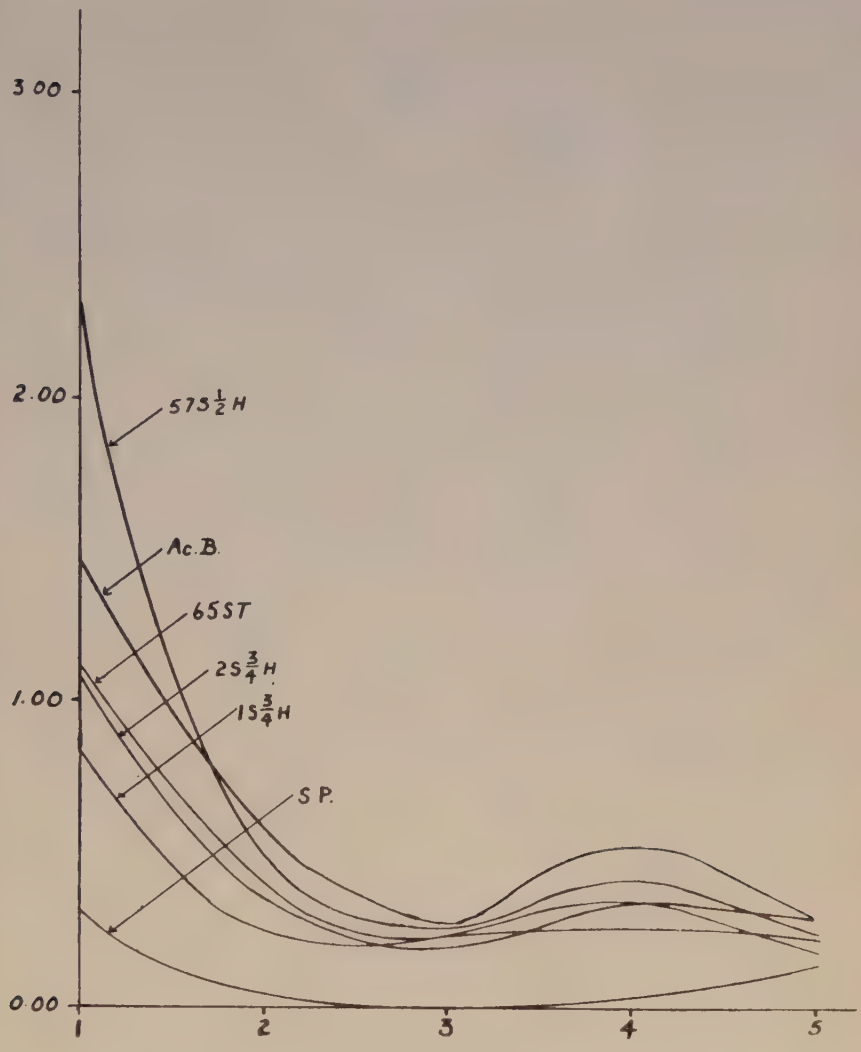

FIGURE 2. Corrosion of aluminium and aluminium alloys by acid cream.

test periods the corrosion rates become relatively constant at a low value indicating that the discs had become protected. This protection is probably due to the development of a corrosion-resistant film on the discs. In general the alloys of greatest purity exhibited the lowest corrosion rates.

In the fourth corrosion test with acid cream (Figure 2) the maxima in the curves may be explained by the fact that the acidity of the cream in this test was higher than in the other tests. In a further series of tests, using milk acidified to varying degrees of acidity with lactic acid, it was found that aluminium was not appreciably attacked until the degree of acidity reached 1.75–2.0 per cent.

From a practical standpoint the action of milk and cream on aluminium and its alloys is negligible.

TABLE 1.—QUANTITY OF IRON PICKED UP BY SWEET WHOLE MILK WHEN STORED IN THREE TYPES OF CANS AT 4° C. AND THE RESULTING FLAVOUR DEVELOPMENT

Number of hours milk was stored in can	Acidity of milk	*Type of can*					
		Aluminium		Tinned-steel (well tinned)		Tinned-steel (partially rusty)	
		p.p.m. iron	Flavour	p.p.m. iron	Flavour	p.p.m. iron	Flavour
	%						
Start of test	0.17	0.11	Clean	0.11	Clean	0.11	Clean
24	0.17	0.15	Clean	0.14	Clean	0.17	Sl. stale
48	0.17	0.15	Clean	0.18	Clean	0.20	Stale
72	0.17	0.14	Sl. stale	0.18	Sl. stale	0.24	Stale
96	0.17	0.13	Stale	0.26	Stale	0.36	Stale
168	0.17	0.12	Stale	0.16	Stale	0.38	Stale

The Effect of Storage of Milk Products in Aluminium, Well Tinned-steel and Partially Rusty Tinned-steel Cans

In order that a milk can may be satisfactory for the transportation and storage of milk products, it must not impair the keeping quality or flavour of the product. This series of storage tests was conducted to determine the effect of the type of storage can on the keeping quality and flavour of the products.

A summary of the results obtained with milk, acid milk and acid cream when stored in the three types of cans is presented in Tables 1, 2, and 3.

The tables indicate that very little aluminium is dissolved by acid milk and acid cream when stored in an aluminium can at room temperatures. Even at the high acidity (0.83 per cent) encountered in the acid milk test, only 1.56 p.p.m. aluminium were dissolved in 48 hours. The quantity of aluminium dissolved by acid cream in 72 hours was less than 1.2 p.p.m. No flavour changes attributable to these increases in aluminium content of the products were evident—the acid and stale flavours being due to high temperatures and long periods of storage.

When the milk products were stored in well tinned-steel cans no deterioration of flavour or keeping quality was noted except that caused by the storage conditions.

In the tests conducted in partially rusty tinned-steel cans, the milk products dissolved appreciable amounts of iron, and flavour changes occurred which were attributable to the increased iron content of the products. In the case of sweet whole milk, the iron content rose from 0.11 to 0.38 p.p.m. during 168 hours of storage. Although a metallic flavour could not be distinguished on the milk, it did become "slightly stale" 48 hours earlier than the same milk when stored in aluminium or well tinned-steel cans. The quickly developed "slightly stale" flavour, however, might have been due to more difficult cleaning of the partially rusty cans.

High acid milk stored in a partially rusty tinned-steel can dissolved over 7 p.p.m. iron in 48 hours. An off-flavour accompanied this increase in iron content, and, although the flavour could not be distinguished as metallic by the panel of judges, such a flavour might have been present but would tend to be masked by the high acid. In any case the off-flavour was attributed to the high iron content.

Cream stored in partially rusty cans was found to be very susceptible to off-flavour development. After only 24 hours' storage, during which time the iron content had increased from 0.96 to 1.74 p.p.m., the cream was graded as "slightly metallic". Within 48 hours the iron content had increased to 3.32 p.p.m. and the cream was graded as "very metallic". After 72 hours' storage and a further increase in iron to 4.90 p.p.m., the cream was graded as "very metallic and oxidized".

The results of these tests show that the quality of sweet whole milk, high acid milk and acid cream is not lowered by storage in aluminium and well tinned-steel cans. The quality of these products, especially cream, is adversely affected by storage in partially rusty tinned-steel cans. Plant operators who encounter metallic flavour difficulties would do well to use only cans containing no exposed iron.

TABLE 2.—QUANTITIES OF IRON AND ALUMINIUM PICKED UP BY ACID MILK WHEN STORED IN THREE TYPES OF CANS AT ROOM TEMPERATURE AND THE RESULTING FLAVOUR DEVELOPMENT

Number of hours milk was stored in can	Acidity of milk	*Type of can*								
		Aluminium			Tinned-steel (well tinned)			Tinned-steel (partially rusty)		
		p.p.m. iron	p.p.m. aluminium	Flavour	p.p.m. iron	p.p.m. aluminium	Flavour	p.p.m. iron	p.p.m. aluminium	Flavour
	%									
Start of test	0.83	0.05	0.84	Clean acid	0.05	0.84	Clean acid	0.05	0.84	Clean acid
24	0.83	0.04	1.4	Clean acid	0.04	0.76	Clean acid	2.84	0.84	Off-flavour but not distinguishable as metallic
48	0.83	0.03	2.4	Clean acid	0.05	0.52	Clean acid	7.28	0.24	Off-flavour but not distinguishable as metallic

TABLE 3.—QUANTITIES OF IRON AND ALUMINIUM PICKED UP BY CREAM WHEN STORED IN THREE TYPES OF CANS AT ROOM TEMPERATURE AND THE RESULTING FLAVOUR DEVELOPMENT

Number of hours cream was stored in can	*Type of can*											
	Aluminium				Tinned-steel (well tinned)				Tinned-steel (partially rusty)			
	Acidity of cream	p.p.m. iron	p.p.m. aluminium	Flavour	Acidity of cream	p.p.m. iron	p.p.m. aluminium	Flavour	Acidity of cream	p.p.m. iron	p.p.m. aluminium	Flavour
	%				%				%			
Start of test	0.21	0.96	< 0.1	Clean	0.21	0.96	< 0.1	Clean	0.21	0.96	< 0.1	Clean
24	0.47	0.84	< 0.1	Clean acid	0.47	0.84	< 0.1	Clean acid	0.47	1.74	—	Sl. metallic
48	0.62	1.25	0.44	Clean acid	0.62	1.16	—	Clean acid	0.64	3.32	< 0.1	Very metallic
72	0.66	1.26	1.2	Clean acid	0.66	1.26	—	Clean acid	0.70	4.90	< 0.1	Very metallic and oxidized

The Corrosion of Aluminium and Tinned-steel by Cleaning and Sterilizing Solutions

Resistance to corrosion, by cleaning and sterilizing solutions, is probably one of the major factors in determining whether a metal is satisfactory for the fabrication of milk cans. Laboratory corrosion tests, although not entirely satisfactory, do give comparative corrosion rates of the different metals under the same set of conditions. These conditions are usually more severe than would be encountered in actual practice. In these corrosion tests all experimental work has concerned fully immersed conditions and no attempt has been made to ascertain the effect of crevice, of water-line attack, or of aeration. The results presented herewith are at least comparative measures of the corrosiveness of the different alkali, acid and chlorine compounds towards aluminium and tinned-steel. Only a very brief summary of the results can be given.

Corrosion tests with alkaline can washing solutions showed that, in general, those solutions which corroded aluminium also corroded tinned-steel. Various alkaline chemical compounds, i.e., sodium carbonate, trisodium phosphate and sodium metasilicate, as well as admixtures of these compounds, were tested. The results showed that sodium carbonate and trisodium phosphate were very corrosive to aluminium and tinned-steel, whereas sodium metasilicate proved to be non-corrosive to both metals. The addition of sodium metasilicate to sodium carbonate and trisodium phosphate inhibited corrosion. Of ten commercial alkaline can washing compounds studied, only two, both of which contained NaOH, were found to be corrosive to aluminium and tinned-steel. The remaining eight commercial compounds were found to be non-corrosive to the metals. Tinned-steel, with the base material exposed, was, in general, attacked to a higher degree than well tinned-steel. Those commercial alkaline can washing compounds which contain sufficient silicates to inhibit corrosion and which do not contain NaOH are non-corrosive to aluminium and tinned-steel.

Corrosion tests using seven commercial acid can washing compounds revealed that aluminium is much more resistant to attack by acid solutions than is tinned-steel—the corrosion rates for aluminium ranging from 3–15 "m.d.d." (mgm. per sq. dec. per day) and those of tinned-steel ranging from 47–216 "m.d.d.". One of the acid compounds caused visible corrosion of tinned-steel.

Corrosion tests using five commercial chlorine preparations, at a strength of 100 p.p.m. available chlorine, showed that only one compound appreciably corroded aluminium. This compound was of the sodium hypochlorite type stabilized with trisodium phosphate. In general the corrosion rates of tinned-steel were higher than the corrosion rates of aluminium. Any exposed base material of the tinned-steel was rapidly attacked during the tests resulting in the formation of rust. Such was the case with four out of the five compounds studied—the exception being the compound which appreciably corroded aluminium. This would indicate that rusty cans are in great danger of attack on the rusty portions if they are chlorinated.

The results of these corrosion tests indicate that no more difficulty, from a corrosion standpoint, should be encountered in the cleaning and sterilizing of aluminium than tinned-steel milk cans.

The Bacteriological Condition of Aluminium and Tinned-steel Milk Cans after the Completion of Cleaning in Commercial Dairy Plants

Milk cans, as free of bacteria as possible, are necessary for the production of high quality milk. In this section an attempt was made to ascertain whether any one type of can was more readily cleaned than other types of cans. The tests were conducted in a milk plant, a creamery, and the College dairy.

Methods of conducting the tests and the types of cans employed in the tests were given earlier. The total number of colonies of bacteria produced per can was used to place each can into one of the following classifications:

(1) 0 — 40,000
(2) 40,000 — 100,000
(3) 100,000 — 200,000
(4) More than 200,000.

Some 1400 cans of all types were examined over a period of 12 months using various washing solutions. Only a very limited discussion of the results obtained can be given.

Except for a very short time when the cans were new (about one month) 12–40 per cent more of the Danish type aluminium cans were able to be placed in the first classification than could the tinned-steel cans containing seams. This same trend, indicating the more complete destruction of bacteria in aluminium cans, was evident also when the strong acid and caseolytic types of colonies were counted and also by the rate of reaction of the can rinsings when inoculated into litmus milk.

Investigations were made in an attempt to account for the apparently more thorough cleaning of the aluminium cans.

In the first place it is argued that, because the aluminium cans are seamless and very smooth internally, they would present little or no protection for bacteria during the cleaning process. On the other hand, tinned-steel cans containing seams and rust spots would offer a certain amount of protection to bacteria during the cleaning process. In the subsequent vigorous rinsing of the cans, to obtain the sample for plating, some of these "protected" bacteria would be removed thus giving rise to the higher counts. Bacteriological examination of a number of old, partially rusty tinned-steel cans, from time to time during this study, revealed that in the great majority of cases they produced over 200,000 colonies per can. In view of the fact that they were treated in a similar manner to the other cans in this study it would appear that seams and rust spots were at least a partial cause of the higher bacterial counts of tinned-steel cans.

Rough seams and rust spots also create a rougher surface in the cans. It seems fairly obvious that milk residues will cling more tenaciously to a rough surface than a smooth one and hence milk residues in a rough surfaced can will be more difficult to remove than those in a smooth surfaced can. During hot weather these milk residues are particularly noticeable and are the cause of more difficult cleaning. Tests conducted in a milk plant during hot weather revealed that about 86 per cent of the Danish type aluminium cans could be placed in the first classification, whereas only 58 per cent of het tinned-steel cans with seams were in the same classification. In the

cumulative collection and storage of cream, the conditions for the development of a milk residue film are ideal because the cream remains in the can for 3–7 days. Tests conducted in a creamery during the hot summer months revealed that 85 per cent of the aluminium cans were able to be placed in the first classification while only 47 per cent of the tinned-steel cans were in that classification.

Because the aluminium cans used in this study have heavy cast iron reinforcement rings around the bases and shoulders and because the body metal of the aluminium cans is thicker than that of the tinned-steel cans, it was reasoned that the aluminium cans might attain and retain a higher temperature during and after washing than would the tinned-steel cans. Thermocouple can temperature measurements revealed, however, that both types of cans attained the same temperatures within the can washers and that these temperatures were never greater than 80° C. This was rather astonishing in view of the fact that steam under pressure is used in the steam jets of the washers. In the authors' opinion much improvement could be made in the washing of milk cans if temperatures of cans inside the washers could be increased. Can temperature measurements taken immediately after the cans were removed from the washers revealed that aluminium cans retained a higher temperature for a longer period of time than did tinned-steel cans. Aluminium cans, 3 or 5 minutes after removal from the washers, were always 3°–7° C. hotter than the tinned-steel cans. This retention of a higher temperature by the aluminium cans could possibly result in more complete destruction of bacteria.

The results of these tests indicate that aluminium cans may be more readily cleaned than tinned-steel cans. It must be remembered that the results presented here were obtained with cans which had been in service for a maximum of 15 months. It is to be expected that results obtained on the same cans after a longer period of service will largely depend upon such factors as mechanical injuries, corrosion, opening of seams, etc. From observations made in the following section it would appear that the aluminium cans will suffer less from the effects of service and therefore it is probable that they will continue to be more easily cleaned than tinned-steel cans.

Further studies should be made using seamless tinned-steel cans. It is probable that they would give better results than the seamed tinned-steel cans used in this study. However, even with the seamless type cans, once the tin wears off and rust spots form, satisfactory cleaning will be hard to accomplish.

The Effect of Commercial Service on Aluminium and Tinned-steel Cans

It is rather difficult to determine just when tinned-steel milk cans are in need of retinning or when they should be discarded. Aluminium cans on the other hand should be serviceable until mechanical injuries inhibit their use. It has already been pointed out that the condition of the milk can has a great effect on the quality and flavour of milk. It seems reasonable, therefore, that only milk cans in excellent condition should be used for the transportation and storage of milk.

In this study an attempt has been made to determine the effect of service on the various types of milk cans. Aluminium and tinned-steel cans were placed in service at various milk plants, and at the time of writing,

had been in service for a period of 15 months. The cans were used for typical farm collection work—one aluminium can being kept with one tinned-steel can as far as possible. Observations were made from time to time during the 15-month period.

The Danish type aluminium cans stood up remarkably well. Rough handling and mechanical dumping equipment caused numerous scratches on the external surfaces but none of these was deep. Internally the Danish cans are still very smooth and no signs of corrosion are in evidence. Because the Danish type cans are fitted with lids without an overhanging lip, damage resulted to the lips of the can necks due to hammering of the lids in removal. This defect was corrected in the Canadian designed aluminium cans. The shoulder protection rings of the Danish type cans are not at the same level as the protection rings of the tinned-steel cans. Therefore, when the two types of cans are in frictional contact, a band of black discoloration develops just beneath the shoulder protection rings of the Danish type cans. This defect was also corrected in the Canadian designed can.

The Canadian type aluminium cans proved to be very resistant to the effects of commercial service. No damage, except a few external scratches caused by mechanical dumping equipment, is evident.

The tinned-steel type of can containing seams proved to be very readily damaged under service conditions. This damage was especially noticeable on the internal surfaces of the cans. Rusting occurred along the seams almost immediately after the cans were placed in service and this rusting became progressively worse as service continued. Rust spots also developed rather quickly on the necks of the cans and on other internal portions of the cans which had been scraped by stirring rods, etc. The tinned-steel cans were also more easily dented than aluminium cans. The shoulder protection rings of the tinned-steel cans were also easily damaged—in one case the ring was detached from the can at the end of 6 months' service and in other cases the rings were loose. The tinned-steel seamless type of can was found to be much more resistant to rusting. However, when rusting did take place it again became progressively worse as service continued.

From the observations made in this study it may be assumed that aluminium cans will have a longer life than tinned-steel cans. However, since this study covered only a relatively short period, no definite conclusions can be made regarding the exact service life of any one type of can.

SUMMARY AND CONCLUSIONS

A comparative study of aluminium and tinned-steel milk cans is presented. These investigations included the determination of the effect of milk and cream on aluminium and its alloys, the effect of storage of milk products in aluminium and tinned-steel cans, the effect of cleaning and sterilizing solutions on aluminium and tinned-steel, the numbers of bacteria remaining in milk cans after commercial cleaning, and the effect of service on aluminium and tinned-steel cans.

A series of corrosion tests showed that aluminium and its alloys are not appreciably corroded by milk and cream. Evidence is presented which indicates that a corrosion-resistant film is built up on aluminium.

The flavour and quality of milk products are not affected by storage in aluminium and well tinned-steel cans. Storage of milk products in partially rusty cans results in deterioration of quality and flavour.

Corrosion tests revealed that most alkaline cleaning compounds which are corrosive to aluminium are also corrosive to tinned-steel. Inhibition of corrosion by alkaline compounds (except NaOH) may be accomplished by the addition of sodium metasilicate. Aluminium is more resistant to corrosion by acid can washing solutions than is tinned-steel. Chlorine solutions, at a strength of 100 p.p.m. available chlorine, were found to be relatively non-corrosive to aluminium unless they were stabilized by trisodium phosphate. Iron (as in rusty cans) is rapidly attacked by chlorine solutions.

Bacteriological examination of 1400 milk cans using a controlled rinse technique revealed that aluminium cans contained relatively fewer numbers of bacteria, after the completion of commercial washing procedures, than did tinned-steel cans.

Evidence is presented which indicates that aluminium cans will have a longer service life than tinned-steel cans.

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